Stability of Materials in Supercritical Ammonia Solutions

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Improvements to the growth of nitride crystals in ammonothermal growth environments can be achieved through improved autoclave designs, purity, and use of *in-situ* monitoring techniques. Given the limited data available on the stability of materials in supercritical ammonia solutions, this study intends to broaden the known dataset by experimentally investigating the mechanical and chemical stability of 35 bulk metals, 2 bulk metalloids, and 17 bulk ceramics while identifying suitable materials for future in-depth corrosion studies. This was performed by exposing each material to three different supercritical ammonia solutions in nickel-chromium superalloy autoclaves held at an external wall temperature of 575 °C for 4—12 days. The solutions were formed by initially filling the autoclave with pure ammonia (NH₃), ammonia and sodium (NH₃-Na), or ammonia and ammonium chloride (NH₃-Cl) to achieve total system pressures of 100—250 MPa. Zirconia, silicon carbide, tungsten carbide, molybdenum and its alloys, tungsten and its alloys, and a cobalt-tungsten-aluminum alloy

(Co₈₀W_{10.6}Al_{9.4}) appeared stable in all three environments. Vanadium, niobium, and tantalum appeared chemically stable in all environments, though these samples embrittled and gained weight. Oxides containing high concentrations of aluminum, silicon, and/or zirconium were most stable in NH₃-Cl while magnesium oxide was not stable in any environment. Silicon nitride corroded in NH₃-Na, yet appeared stable in NH₃ and NH₃-Cl. Silver and a platinum-ruthenium alloy (Pt₉₁Ru₉) appeared to be the only noble metals stable in NH₃-Na while all of the noble metals investigated, except for silver, appeared to be stable in NH₃-Cl. Copper and its alloys were not stable in any environment except for Constantan (Cu₅₃Ni₄₇) in NH₃. Exposed thermocouples of type K and N remained functional post-run in NH₃ and NH₃-Na. Iron and nickel and their respective alloys appeared stable in NH₃ and NH₃-Na but not in NH₃-Cl. Most other metals and metalloids investigated in this study were not stable in any of the three solutions and lost weight. Results in this study agree with literature data, where available.

Keywords: corrosion, supercritical ammonia, ammonothermal, stability of materials

Graphical Abstract:

1. Introduction

Supercritical ammonia solutions provide a suitable environment for the growth of nitride crystals.^{1,2,3} Under conditions of high temperature and pressure, this process may also be referred to as the ammonothermal method and is an analogue to the well-established hydrothermal method for oxide crystal growth.

Of current interest is the ammonothermal growth of single crystal gallium nitride (GaN)^{4,5}, a wide bandgap semiconductor that is predominately used for the fabrication of optoelectronic devices such as blue and green light emitting diodes (LEDs) and lasers. Typical growth temperatures range from 450 °C to 650 °C and total system pressures from 100 MPa to 300 MPa. In the case of GaN growth, a mineralizer needs to be added to pure ammonia to provide any noticeable solubility of gallium compounds in the solution. Alkali metals, commonly sodium or potassium, are added to yield a basic solution^{6,7} while halogen compounds, commonly fluorides or chlorides, are added to yield an acidic solution⁸. The resulting growth environment contains high concentrations of ammonia, hydrogen, and nitrogen along with low concentrations of dissolved mineralizers and gallium-containing compounds. Molecular hydrogen and nitrogen gases are primarily formed by the decomposition of ammonia.

Containing the corrosive growth environment at both elevated temperatures and pressures requires the use of autoclaves made of high temperature, high strength materials. The current generation of autoclaves are typically made of nickel-chromium based superalloys such as Inconel 718 and René 41. While these alloys provide sufficient corrosion resistance for basic supercritical ammonia solutions, they are ill-suited for acidic growth environments due to substantial etching of the autoclave material.^{9,10} Inconel 625 provides improved corrosion resistance but is not recommended for long-term use. A partial solution to this dilemma is provided by using a corrosion resistant liner such as platinum,¹¹ though this introduces design challenges and a significant increase in equipment cost.

Nitride crystal growth in basic and acidic supercritical ammonia solutions could greatly benefit from an improved understanding of the stability of materials in these environments. Some work has been performed on this topic, yet there is currently only a limited number of dedicated

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4 experiments performed on materials exposed to anhydrous, supercritical ammonia under
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6 ammonothermal conditions to determine their stability^{1,9,10,12,13}, especially when comparing to
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8 the well explored hydrothermal system.¹⁴ An expansion to this dataset would permit exploring
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10 new autoclave materials, less-expensive liner options, novel materials for use as internal
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12 components in the growth environment, unique *in-situ* monitoring techniques, and soluble
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14 materials for modifying solution chemistry.
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20 In order to significantly expand the currently-limited number of materials whose stability has
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22 been studied in supercritical ammonia solutions at elevated temperatures, this paper surveys and
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24 comments on the observed stability of 35 bulk metals, 2 bulk metalloids, and 17 bulk ceramics
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26 (oxides, nitrides, and carbides) in three different supercritical ammonia solutions. The solutions
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28 were formed by filling autoclaves with ammonia (neutral), ammonia and sodium
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30 (ammonobasic), or ammonia and ammonium chloride (ammonoacidic). The autoclave is then
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32 heated to a target external wall temperature of 575 °C thereby generating pressures of 80—260
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34 MPa once the system equilibrated. Our recent studies show that for these run conditions, 10—
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36 30% of the initial ammonia filled into the system may decompose into molecular hydrogen and
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38 nitrogen.¹⁵ The chemical and mechanical stability of the investigated materials was determined
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40 from changes in the sample weight, thickness, mechanical behavior (cracking, embrittlement),
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42 surface behavior (pitting, spalling, powder formation), and coloration. Changes in the chemical
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44 composition of select samples was analyzed by energy-dispersive X-ray spectroscopy (EDX).
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49 The materials were classified according to their applicability and potential engineering use with
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51 supercritical ammonia solutions. The main purpose of this study was to screen a large set of
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53 materials and identify a subset of potential materials for future in-depth studies.
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2. Experimental Methods

A summary of the materials investigated in this study is provided in Table 1. These materials were selected for a variety of reasons including covering most major classes of ceramics and structural metal alloys, ease of availability, low cost, and possible use for *in-situ* techniques. Additionally, high purity materials were selected for identification as possible solutes or for use as liner materials or internal components.

Table 1

Summary of materials surveyed for their stability in supercritical ammonia solutions. Detailed information on the materials, including their nominal chemical composition, can be found in Table A.2.

Class	Subclass	Common Material Name*
Bulk Ceramic	Oxide	Alumina Silicate, Fused Silica, Glass Ceramic, Glass
		Mica, Machinable High-Alumina Ceramic, Magnesium
		Oxide, Quartz, Sapphire, Sintered Alumina, Soda Lime
		Glass, Yttria-stabilized Zirconia, Zirconia
	Nitride	Hot-pressed Boron Nitride, Pyrolytic Boron Nitride,
		Silicon Nitride
	Carbide	Silicon Carbide, Tungsten Carbide
Bulk Metalloid	Pure	Germanium, Silicon

Bulk Metal	Pure	Aluminum, Cobalt, Copper, Gold, Iridium, Lanthanum, Magnesium, Molybdenum, Nickel, Niobium, Palladium, Platinum (TC R-), Scandium, Silver, Tantalum, Titanium, Tungsten, Vanadium, Yttrium, Zirconium
	Cobalt Alloys	Cobalt-Tungsten-Aluminum Alloy
	Copper Alloys	Brass 260, Constantan (TC J-, TC E-)
	Iron Alloys	1018 Steel, 15-5 PH, 17-4 PH, 316L Stainless Steel
	Molybdenum Alloys	Titanium-Zirconium-Molybdenum
	Nickel Alloys	Chromel C (TC K+), Alumel (TC K-), Hastelloy C-276, Nicrosil (TC N+), Nisil (TC N-)
	Platinum Alloys	Platinum-Rhodium (TC R+), Platinum-Ruthenium
	Tungsten Alloys	High-Strength Durable Tungsten, Tungsten-Rhenium (TC C-), Tungsten-Rhenium (TC C+)

* Abbreviations: TC = Thermocouple, +/- indicates positive or negative lead for indicated thermocouple type.

The stability of these materials in supercritical ammonia solutions was determined according to the following procedure. Materials were sourced from various vendors and reduced in size, as specified in Table A.2, to fit inside a one inch inner diameter autoclave. Samples were cleaned sequentially in deionized (DI) water, acetone, and isopropyl alcohol (IPA) for at least one minute each using an ultrasonic bath. After drying, their weight was measured using a scale (Manufacture: Mettler Toledo, AB135-S/FACT, Precision: ± 0.01 mg) and their dimensions measured using a micrometer (Manufacture: Mitutoyo, No. 293-765-30, Accuracy: ± 1 μ m), an

absolute digital indicator (Manufacture: Mitutoyo, Model: ID-C112E, Accuracy: $\pm 5 \mu\text{m}$), or a caliper (Manufacture: Mitutoyo, Model: CD-6" ASX, Accuracy: $\pm 25 \mu\text{m}$). An optical image was taken using a camera (Manufacture: Canon, Model: PowerShot SD780 IS). In some cases, optical micrographs were taken (Manufacture: Nikon, Model: Eclipse L200).

One or more samples were then placed inside an empty nickel-chromium superalloy autoclave with one inch inner diameter and internal volume of approximately 65 ml. A René 41 reactor was used for supercritical ammonia solutions starting with ammonia and sodium ($\text{NH}_3\text{-Na}$) while an unlined Inconel 625 autoclave was used for solutions starting with ammonium chloride ($\text{NH}_3\text{-Cl}$) due to the unacceptably high etch rate of the René 41 autoclaves in the $\text{NH}_3\text{-Cl}$ environment. For solutions starting with pure ammonia (NH_3), either a René 41 or an unlined Inconel 625 autoclave was used.

The addition of either sodium (Source: Alfa Aesar, 99.95 % metal basis purity) or as received ammonium chloride powder (Source: Alfa Aesar, 99.999 % purity) to the autoclave was performed inside a nitrogen glove box (Manufacture: MBraun, Model: Labmaster 130) containing low oxygen and water concentrations ($\leq 1 \text{ ppm}$) after the samples were placed inside the autoclave. The sodium cubes were prepared on a polytetrafluoroethylene (PTFE) sheet using a stainless steel knife. The sodium was removed from the mineral oil used to store it and then all surfaces that came into contact with the mineral oil were cut off. The autoclave was then sealed with a commercially pure nickel gasket (Ni 200/201, >99 % Purity) and a head assembly that contained a pressure transducer (Manufacture: Omegadyne, Model: PX02, $207 \pm 2 \text{ MPa}$ or Manufacture: Honeywell, Model: HP, Span: $414 \pm 4 \text{ MPa}$), a rupture disc and a needle valve. The entire autoclave assembly was then weighed (Manufacture: A&D, GX-10k, Precision: ± 10

mg) and connected to the ammonia filling station. A detailed overview of the ammonia filling station can be found in Ref [16].

The autoclave was evacuated and underwent multiple pump-purge cycles using ultra high purity nitrogen. After the final cycle the autoclave was fully evacuated and cooled using liquid nitrogen. Once sufficiently cooled, a predetermined amount of gaseous ammonia was allowed to flow into the autoclave and liquefy. The amount of ammonia was selected to target a pressure of 250 MPa for René 41 autoclaves and 100 MPa for Inconel 625 autoclaves. After filling, the autoclave was allowed to warm to room temperature, weighed on the same scale as before, and then transferred into a resistive heater setup. Figure 1 shows a schematic of the experimental setup and a loaded autoclave in the resistive heater as it would exist in the corrosion runs. Type K thermocouples with special limits of error were used to measure the external wall temperature of the autoclave.

Figure 1 Schematic of the experimental setup using a Ni-Cr superalloy autoclave (11) sealed using a nickel gasket (12), a needle valve made of 316 stainless steel (SS 316) (13), a rupture

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4 disc made of SS 316 (14), and a pressure transducer (42) which is heated externally using
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6 ceramic insulated resistive heaters (21). Copper heat spreaders (22), centering rings (23), and
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8 thermal insulation (24) are used to improve temperature uniformity which is measured on the
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10 external surface of the autoclave using copper tipped (25) type K thermocouples (41). Samples
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12 (31) are situated at the bottom of the one inch inner diameter autoclave.
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20 The autoclave was heated at a rate of 2 °C/min to an external wall temperature of approximately
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22 575 °C and was held at that temperature for 4—12 days. Later runs performed in the same
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24 experimental setup for a different study using an internal thermocouple suggest that the actual
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26 fluid temperature is approximately 80 °C lower than the measured external wall temperature. The
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28 temperature of the autoclave was lowered during the run in a few cases where the pressure
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30 exceeded the safe values of 260 MPa or 110 MPa for the René 41 or Inconel 625 autoclaves,
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32 respectively. Upon successful conclusion of the run, the autoclave was removed from the heater
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34 setup, weighed, vented, and weighed again. A summary of all runs along with the fill amounts of
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36 ammonia, sodium or ammonium chloride, and other run attributes (temperature, peak pressure,
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38 and duration) for all runs can be found in Table A.1.
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45 After unsealing the autoclave the system was cleaned. In the case of sodium containing runs, the
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47 system was first neutralized (dissolution of pyrophoric materials) using an IPA-water mixture to
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49 remove all traces of sodium-containing compounds. The samples were removed from the
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51 autoclave, then cleaned in an ultrasonic bath using DI water. Characterization consisted of
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53 measuring the weight and size of the sample, noting their visual appearance, and taking pictures
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55 of the sample. Additionally, some samples were stressed by hand to determine changes in their
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57 mechanical behavior.
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4 To minimize run-to-run contamination for the five autoclaves used in this study (two René 41
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6 and three Inconel 625 autoclaves), the autoclaves were extensively cleaned after each run by
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8 removing deposition and/or corroded surfaces using silicon carbide and aluminum oxide
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10 abrasives, followed by cleaning using acetone, IPA, and DI water in an ultrasonic bath. In some
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12 instances machining or drilling was required to remove metallic deposits which were present on
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14 the inner surfaces of the autoclave or nozzle. While every effort was made to remove
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16 contaminants prior to a new run, only visual inspection was performed to verify that the inner
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18 surfaces of the autoclave and nozzle were clean.
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24 To minimize the number of runs and maximize the number of materials that could be explored,
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26 samples expected to behave similarly were sometimes co-loaded. Co-loading was performed by
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28 simply placing two or more samples inside and on the bottom of an autoclave. No attempt was
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30 made to individually separate samples to ensure that they did not touch during the run.
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35 Select materials that demonstrated stability in $\text{NH}_3\text{-Cl}$ environments while using an unlined
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37 Inconel 625 autoclave with nickel gasket (titanium, Hastelloy C-276, aluminum oxide, alumina
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39 silicate, and fused silica) were re-run in a capsule configuration to minimize contamination from
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41 the autoclave walls and nickel gasket. Capsules were made out of the desired material either in
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43 the form of a close ended tube and a slip-fit plug for the top, or an open ended tube with two slip-
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45 fit plugs for both ends. A single capsule per run was placed inside an unlined Inconel 625
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47 autoclave and ammonium chloride powder was placed in the capsule and loosely sealed by
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49 placing the plug on the top. While this did not prevent ammonium chloride from interacting with
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51 the wall or gasket, it did appreciably increase the time the desired material was exposed to an
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53 uncontaminated $\text{NH}_3\text{-Cl}$ environment.
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Table A.2 provides a detailed summary of all the samples examined in this study including the approximate initial size (within 1 mm), form, and weight before and after the run. The sample number is created by merging the run number (six numeric digits followed by a letter signifying a particular autoclave) provided in Table A.1 with an underscore, the letter A, and a numeric digit starting from one and incremented by one for each additional sample placed in the same autoclave and run. Samples with identical run numbers were co-loaded in the same run.

3. Results and Discussion

Results from the various runs will be summarized and presented as follows. Materials will be grouped by class (bulk ceramic or bulk metal) and subclass (oxide, nitride, carbide, or the various metal alloy systems). The results from the two bulk metalloid samples will be presented under the pure bulk metal class.

We report sample analysis based on their chemical and mechanical stability in each of the three solutions and a summary of notable observations specific to each material will be reported. The chemical stability of materials was determined by their relative weight change (raw data is provided in Table A.2), thickness change (when accurately obtained with sufficiently small experimental error), and color change. In some cases EDX was also used to determine chemical stability. Chemical stability is defined here as the absence of any measurable chemical changes that compromised the integrity of the sample. For example, a sample can change color and lose a small amount of mass but still be considered chemically stable if the changes are such that the expected lifetime of the sample would be long compared to the length of a typical ammonothermal run. The mechanical stability of materials was determined by observable

changes in mechanical properties (such as embrittlement) and macroscopic defects (such as crack formation, spalling, etc.). When the general term ‘stable’ or ‘unstable’ is used to describe a sample, it refers to both chemical and mechanical stability. Should a sample only exhibit one kind of stability, the type of stability is explicitly stated (‘chemically stable’ and ‘mechanically stable’).

A general recommendation is made for each material in each solution based on the aggregate knowledge collected for the material and is presented using a checkmark (✓), a circle (○), or a cross (×) at the end of each subclass summary section.

A checkmark indicates that the material has good to excellent chemical and mechanical stability in the indicated supercritical ammonia solution and should be considered for future in-depth studies for chemically inert components (for example, internal components, liners, etc.). A circle indicates that the material could potentially be used under certain circumstances, though it should not necessarily be considered simultaneously chemically and mechanically stable. A cross suggests the material is not stable for the given environment as it severely degraded, though it may be suitable as a solute.

It is important to emphasize that the recommendations in this study are based on short term exposure (< 300 hr) in nickel-chromium superalloy autoclaves with stainless steel head assemblies and commercially pure nickel gaskets. Galvanic protection of samples through the reactor alloys, gasket, head assembly, or any co-loaded samples cannot be ruled out as these materials are not guaranteed to be stable. Furthermore, the nickel gasket and autoclave walls experienced significant etching for all NH₃-Cl runs and the dissolved metals appeared to have been re-deposited at the bottom of the autoclave, presumably the hottest location in the autoclave. The contribution of the dissolved autoclave metals (primarily nickel and chromium) to

the apparent stability (or instability) of materials in $\text{NH}_3\text{-Cl}$ is unknown. However, the effect of this uncontrolled contamination was reduced for runs performed using capsules, thereby effectively creating a higher purity environment within the capsule.

It should also be noted that all samples were analyzed post-run and after exposing the samples to water. In some cases the water reacted with the samples, possibly by oxidation of species that were present on the surface. Furthermore, most $\text{NH}_3\text{-Cl}$ runs exhibited pronounced rust-colored deposition post-run. The deposition washed off in most cases, but not all. The remnants of the deposition can be seen in select post-run photographs. EDX analysis of these deposits suggested that they contained a chromium chloride (chromium to chlorine ratio was 1:2.5), the metal presumably originating from the autoclave walls (Inconel 625) or the head assembly (316 SS).

For convenience, all material compositions are provided and presented in atomic %. Most industrial metal alloys and non-stoichiometric oxides are commonly referenced by weight %, so these samples have been converted to atomic % for consistency. The reader is referred to Table A.2 for details on each sample.

3.1. Stability of Bulk Ceramics

3.1.1. Oxide Ceramics

Summary — Overall, oxide ceramics were found to be unstable in $\text{NH}_3\text{-Na}$ environments. A notable exception is pure zirconia, which exhibited very limited corrosion and no change in mechanical behavior after a 6 day exposure at 575 °C when co-loaded with glass ceramic and machinable high-alumina ceramic. Oxide ceramics were also found to be generally stable in NH_3

environments. Oxides with a large number of different cations were more likely to display changes in optical properties, though no significant changes in observed mechanical properties were found. Certain oxide ceramics appeared to be stable in $\text{NH}_3\text{-Cl}$ solutions. Those with high alumina, silica, and zirconia contents provided the highest stability. Alumina, silica, alumina silicate, zirconia, and 8 mol% yttria stabilized zirconia (YSZ) were found to be chemically stable with occasional, minor discoloration and no observed change in mechanical properties. This is consistent with experiments reported in the literature when exposing quartz¹⁷ and sapphire¹⁸ to supercritical ammonia solutions. Deposition from the $\text{NH}_3\text{-Cl}$ solution occurred on most samples, though this is believed to be from etching of the Inconel 625 autoclave, nickel gasket, or 316 stainless steel head assembly parts. A summary of the recommendations is provided in Table 2.

Table 2

Summary of bulk oxide ceramic samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$
Aluminum Silicate	×	○	✓	-1.62	-0.49	+0.11	130702C_A1	130618G_A1	130521E_A1
Fused Silica	×	✓	✓	-100.00	-0.02	-0.90	130702C_A3	130618G_A3	130521E_A3
Glass Ceramic	×	✓	○	-21.58	+0.02	-0.07	130709D_A1	130611G_A1	130528F_A1
Glass Mica	×	○	○	-4.61	+0.05	-0.44	130702C_A2	130618G_A2	130521E_A2
Machinable High-Alumina Ceramic	×	✓	○	-92.53	-0.89	+4.60	130709D_A2	130611G_A2	130528F_A2

Magnesium Oxide	×	✓	×	-4.05	N/A	-100.00	130425C_A1	130528G_A1	130604E_A1
Quartz	×	✓	✓	-100.00	-0.13	0.00	130412C_A1	130528G_A4	130321G_A1
Sapphire	×	✓	✓	-51.82	0.00	-0.01	130222C_A1	130311C_A1	130312E_A1
				-47.14	-0.03	0.00	130222C_A2	130311C_A2	130312E_A2
Sintered Alumina	×	✓	✓	-4.07	-0.05	-0.04	130222D_A1	130311C_A3	130312E_A3
Soda lime	×	○	○	-100.00	N/A	-1.43	130412C_A2	130528G_A3	130321G_A2
Yttria-stabilized Zirconia	○	✓	✓	-0.05	-0.04	-0.30	130425C_A2	130528G_A2	130604E_A2
Zirconia	✓	✓	○	-0.01	0.00	-0.10	130709D_A3	130611G_A3	130528F_A3

Alumina Silicate ($\text{Al}_{13.1}\text{Si}_{21.0}\text{Fe}_{0.8}\text{K}_{0.6}\text{Ti}_{0.4}\text{O}_{64.1}$) — The alumina silicate samples appeared stable in both $\text{NH}_3\text{-Cl}$ and NH_3 environments. A slight increase in thickness (+20 μm , +0.3 %) and change in coloration from black to dark gray occurred for the NH_3 sample but no degradation in overall mechanical properties was observed. Subsequent capsule testing in $\text{NH}_3\text{-Cl}$ indicated no significant change in properties, though there was some green and yellow deposition. Under $\text{NH}_3\text{-Na}$ conditions the sample severely deteriorated by swelling (thickness increase: +740 μm , +12 %), cracking, spalling and disintegrating into powder on the exposed surfaces of the material. The color changed from black to light gray. (See also Figure 2)

Figure 2 Representative aluminum silicate samples: optical images (a) pre-run, and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*possible uses*, $\circ$), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, $\checkmark$). One square corresponds to 1 mm x 1 mm.

Fused Silica (SiO_2) — The fused silica samples were found to be stable in $\text{NH}_3\text{-Cl}$ and NH_3 environments. The slight weight loss of the $\text{NH}_3\text{-Cl}$ sample may have been due to an edge chipping off during post-run handling. Interestingly, both samples showed varying amounts of small ($\sim 10\ \mu\text{m}$ diameter), shallow protrusions ($\sim 8\text{--}17\ \mu\text{m}$ depth) on the surface of the samples post run. The concentration of the spots was on the order of $10^3\text{--}10^4\ \text{spots}/\text{cm}^2$. No decrease in transparency or clarity of the samples was found. Subsequent capsule testing in $\text{NH}_3\text{-Cl}$ revealed no significant change except for a slight rust colored deposition on all exterior surfaces which was not water soluble. No cracking or structural changes were present. The $\text{NH}_3\text{-Na}$ sample completely disintegrated leaving behind a small amount of white powder which completely dissolved in water. The instability of “supremax” (borosilicate) glass under basic conditions has previously been observed in literature¹ suggesting high silica content glass is generally unstable under basic conditions. (See also Figure 3)

Figure 3 Representative fused silica samples: optical images (a) pre-run and post-run in (b) NH₃ (good to excellent potential, ✓) or (c) NH₃-Cl (good to excellent potential, ✓). One square corresponds to 1 mm x 1 mm.

Glass Ceramic (Si_{20.7}Al_{7.9}Li_{3.2}Mg_{1.9}Ca_{1.4}Na_{0.6}Ti_{0.6}Zn_{0.6}Ba_{0.5}K_{0.4}Zr_{0.4}O_{61.8}) — This material exhibited changes in all environments but had the greatest stability in NH₃ conditions. No changes in thickness or overall mechanical properties were observed. However, short internal cracks formed. The color of the sample changed from pale brown to a uniform, dark orange color. The NH₃-Cl sample etched slightly on the rough sides (thickness change -15 to -20 μm, -0.2 %) and exhibited pronounced internal cracking throughout the sample in the form of long continuous cracks. The color changed from pale brown to dark brown. The NH₃-Na sample severely corroded with significant pitting, surface roughening, cracking, and etching (thickness change: > -600 μm, -12 %). The surface of the sample changed to a dark gray-green crust which was no longer transparent to visible light. Compared to the alumina silicate sample, this sample did not show as much flaking or spalling nor did it yield any powder from its surfaces. (See also Figure 4)

Figure 4 Representative glass ceramic samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Glass Mica ($\text{Si}_{15.4}\text{Mg}_{8.5}\text{F}_{4.2}\text{Al}_{3.2}\text{K}_{2.1}\text{B}_{2.0}\text{O}_{64.5}$) — The bulk of the glass mica underwent a color change from white to a dull gray-tan for runs performed in all three environments. In the $\text{NH}_3\text{-Na}$ environment, the sample lost mass by significantly etching and shedding part of its original surface. The $\text{NH}_3\text{-Cl}$ sample exhibited rust-colored stains on its surface which remained even after mechanical scrubbing. The NH_3 sample increased in thickness (+20 μm , +0.3 %) while also gaining weight. Overall, the mechanical properties of all three samples did not appear to be altered. (See also Figure 5)

Figure 5 Representative glass mica samples: optical images (a) pre-run and post-run in (b) NH_3 -Na (*not recommended*, $\times$), (c) NH_3 (*possible uses*, $\circ$), or (d) NH_3 -Cl (*possible uses*, $\circ$). One square corresponds to 1 mm x 1 mm.

Machinable High-Alumina Ceramic (Al_2O_3) — This material was tested in its unfired and hence unsintered state. Despite this, the NH_3 sample exhibited no noticeable changes. The NH_3 -Cl environment sample had comparable mechanical properties, yet exhibited strong rust coloration on its surface which partially flake off if agitated. This material is not mechanically stable in NH_3 -Na environments as the sample crumbled post-run. (See also Figure 6)

Figure 6 Representative machinable high alumina ceramic samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*good to excellent potential*, $\checkmark$), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, $\circ$). One square corresponds to 1 mm x 1 mm.

Magnesium Oxide (MgO) — Under $\text{NH}_3\text{-Cl}$ conditions this material completely dissolved. Under $\text{NH}_3\text{-Na}$ conditions the material etched only slightly, resulting in crystallographic pitting and grooving of the surfaces (possibly along grain boundaries) with an overall thickness change of $-5\text{ }\mu\text{m}$ (-1%). When exposed to a pure NH_3 solution the sample exhibited neither a change in optical or mechanical properties. However, a small amount of the co-loaded soda lime glass sample adhered to the magnesium oxide, making it impossible to determine any weight change. No measureable thickness change occurred and no pits or other surface deterioration were detected using differential interference contrast microscopy (DICM), suggesting no etching occurred. (See also Figure 7)

Figure 7 Representative magnesium oxide samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✗) or (c) NH_3 (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Quartz (SiO_2) — Single crystal silicon dioxide appears to be stable in NH_3 and $\text{NH}_3\text{-Cl}$ environments, as indicated in prior in literature.^{17,19} No corrosion or changes in sample properties were detected for the NH_3 sample while the $\text{NH}_3\text{-Cl}$ exhibited a slight coloration due to deposits on the surface of the sample. In the $\text{NH}_3\text{-Na}$ environment the sample completely disintegrated. (See also Figure 8)

Figure 8 Representative quartz samples: optical images (a) pre-run and post-run in (b) NH_3 (*good to excellent potential*, ✓) or (c) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Sapphire ($\alpha\text{-Al}_2\text{O}_3$) — Single crystal alumina wafers with a large, nominally c -plane orientated surface appeared to be stable in both NH_3 and $\text{NH}_3\text{-Cl}$ environments, exhibiting no changes in

properties or appearance. This observation is comparable to literature on $\text{NH}_3\text{-Cl}$ exposure to sapphire used as a pressure-retaining window for *in-situ* monitoring of the supercritical fluid.¹⁸ In the $\text{NH}_3\text{-Na}$ environment the material corroded non-uniformly, primarily by thinning and formation of macroscopic holes throughout the sample. The remaining planar surfaces were analyzed using DICM and crystallographic faceting was found, suggesting an anisotropic etch. (See also Figure 9)

Figure 9 Representative sapphire samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Sintered Alumina (Al_2O_3) — Sintered, polycrystalline alumina appears to be stable in NH_3 and $\text{NH}_3\text{-Cl}$ environments. Some black to dark rust-colored deposition occurred on the $\text{NH}_3\text{-Cl}$ sample. Subsequent capsule testing in $\text{NH}_3\text{-Cl}$ showed no significant structural changes or

discoloration with the exception of several black streaks. Slight etching may have occurred as evidenced by mass loss in the capsule test (-0.83 mg, -0.005 %). In the $\text{NH}_3\text{-Na}$ environment the sample retained its structural integrity, though this sample was also thicker (2.3 mm) when compared to the chemically identical sapphire sample (0.3 mm). It exhibited a comparable amount of mass loss to the sapphire samples (~ 100 mg, or ~ 39 μm for this sample). (See also Figure 10)

Figure 10 Representative sintered alumina samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*good to excellent potential*, $\checkmark$), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, $\checkmark$). One square corresponds to 1 mm x 1 mm.

Soda lime ($\text{Si}_{24.4}\text{Al}_{0.5}\text{K}_{0.6}\text{Mg}_{2.3}\text{Na}_{10.0}\text{Ca}_{2.5}\text{O}_{59.7}$) — Soda lime glass is not stable in the $\text{NH}_3\text{-Na}$ environment and completely disintegrated. In the NH_3 environment it appeared to be stable, though its color changed to a dark red. Furthermore, a part of the sample adhered to the co-

loaded magnesium oxide sample which made it impossible to determine the weight changes of either sample. Nevertheless, only small pits were observed on the surface post-run and no significant change in thickness ($< 3 \mu\text{m}$, $< 0.3 \%$) was measured. The $\text{NH}_3\text{-Cl}$ sample exhibited a dark red discoloration while also losing mass and forming a wavy surface on a macroscopic scale ($> 1 \text{ mm}$). (See also Figure 11)

Figure 11 Representative soda lime samples: optical images (a) pre-run and post-run in (b) NH_3 (possible uses, O) or (c) $\text{NH}_3\text{-Cl}$ (possible uses, O). One square corresponds to $1 \text{ mm} \times 1 \text{ mm}$.

Yttria-stabilized Zirconia ($\text{Zr}_{29}\text{Y}_5\text{O}_{66}$) — Single crystal, 8 mol% yttria-stabilized zirconia appears to be stable in both NH_3 and $\text{NH}_3\text{-Cl}$ environments. The minor weight loss that was observed can be attributed to macroscopic mass loss at the edges during handling. Some deposition occurred on the $\text{NH}_3\text{-Cl}$ sample. The $\text{NH}_3\text{-Na}$ sample did not lose appreciable mass and did not change measurably in thickness. The originally specular surface of the sample became diffuse, indicating slight etching. DICM micrographs revealed crystallographic features, suggesting an anisotropic etch. (See also Figure 12)

Figure 12 Representative yttria-stabilized-zirconia samples: optical images (a) pre-run, and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (c) NH_3 (*good to excellent potential*, $\checkmark$), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, $\checkmark$). One square corresponds to 1 mm x 1 mm.

Zirconia (ZrO_2) — This material exhibited some level of stability in all three solutions. While the $\text{NH}_3\text{-Na}$ sample changed in color (from white to pale yellow), it did not appear to have gained mass or increased in thickness. The $\text{NH}_3\text{-Cl}$ sample changed color from white to purple. It also showed slight mass loss (-9.6 mg, -0.1 %) and increased in diameter ($\sim +20\ \mu\text{m}$, +0.3 %). None of the samples exhibited a change in observed mechanical behavior. Literature reports attempts of using zirconia as a coating on Inconel 718, which resulted in the complete loss of the coating, most likely due to poor adhesion on the Inconel substrate.¹² (See also Figure 13)

Figure 13 Representative zirconia samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

3.1.2. Nitride Ceramics

Summary — Boron nitride appeared to be slightly soluble in all three solutions with the highest deterioration of the sample occurring in the $\text{NH}_3\text{-Na}$ environment. Both kinds of boron nitride (hot-pressed and pyrolytic) changed from white to yellow in color in the $\text{NH}_3\text{-Cl}$ environment. All of the pyrolytic boron nitride samples experienced pronounced post-run flaking of material off of the surfaces. Silicon nitride appeared to be stable in NH_3 and $\text{NH}_3\text{-Cl}$ solutions with no observable changes due to exposure to these environments. A summary of the recommendations is provided in Table 3.

Table 3

Summary of bulk nitride ceramic samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Hot-Pressed Boron Nitride	×	○	○	-5.51	-0.54	-0.20	130321C_A2	130425D_A1	130416G_A4
Pyrolytic Boron Nitride	×	○	○	-19.77	-1.55	+0.84	130321C_A1	130425D_A2	130416G_A3
Silicon Nitride	×	✓	✓	-1.56	-0.02	-0.02	130321C_A3	130425D_A3	130416G_A2

Hot-Pressed Boron Nitride (BN) — All samples experienced slight mass loss. The NH₃-Na sample experienced some flaking along with a thickness change of ~ -30 μm (~ -2 %), though the sample remained structurally sound. Additionally, the originally matte finish surface became highly reflective after the runs. This suggests the formation of smooth facets and possibly an anisotropic, crystallographic etch. The sample exposed to the NH₃ environment only experienced a slight thickness reduction (-2 μm, -0.2 %) and weight loss (-2.40 mg). The rust-colored deposits on the NH₃-Cl sample could not be mechanically removed. No measurable change in thickness occurred for this sample though slight mass loss occurred, which is comparable to data presented in literature.¹² (See also Figure 14)

Figure 14 Representative hot-pressed boron nitride samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*possible uses*, $\circ$), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, $\circ$). One square corresponds to 1 mm x 1 mm.

Pyrolytic Boron Nitride (BN) — Compared to the hot-pressed boron nitride, all samples thinned to a greater extent and experienced flaking. The $\text{NH}_3\text{-Na}$ sample easily broke into smaller pieces while showing black coloration on spots where flakes of BN were removed. The NH_3 sample seemed to have etched uniformly and changed in thickness by $-19\ \mu\text{m}$ (-2%). The $\text{NH}_3\text{-Cl}$ exhibited a slight weight gain (possibly due to rust-colored depositions) despite changing in thickness by $-9\ \mu\text{m}$ (-1%), suggesting it is not entirely chemically stable in $\text{NH}_3\text{-Cl}$ environments. (See also Figure 15)

Figure 15 Representative pyrolytic boron nitride samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*possible uses*, $\circ$), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, $\circ$). One square corresponds to 1 mm x 1 mm.

Silicon Nitride (Si_3N_4) —Neglecting the rust-colored deposition from the $\text{NH}_3\text{-Cl}$ environment, the silicon nitride samples behaved identically in NH_3 and $\text{NH}_3\text{-Cl}$, being stable in both and not exhibiting any deterioration in structural quality. The mass change of the $\text{NH}_3\text{-Cl}$ sample is in agreement with literature.¹² The $\text{NH}_3\text{-Na}$ sample did not fare as well and experienced a change in thickness of $-24\text{ }\mu\text{m}$ (-0.8%) while displaying a partial crack through the sample. The sample also turned a lighter gray color, similar to the alumina silicate sample. These results are in line with observations made when using high surface area Si_3N_4 under $\text{NH}_3\text{-Na}$ conditions, resulting in the formation of NaSi_2N_3 .²⁰ (See also Figure 16)

Figure 16 Representative silicon nitride samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*good to excellent potential*, $\checkmark$), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, $\checkmark$). One square corresponds to 1 mm x 1 mm.

3.1.3. Carbide Ceramics

Summary — Generally speaking, the investigated carbides appeared to be stable in all three supercritical ammonia solutions. No observable changes in mechanical properties occurred for any of the samples. A slight roughening of silicon carbide in the $\text{NH}_3\text{-Na}$ environment occurred, suggesting slight solubility while the tungsten carbide sample experienced a slight increase in diameter ($\sim +5 \mu\text{m}$, $\sim +0.08 \%$) in $\text{NH}_3\text{-Cl}$ and NH_3 environments. A summary of the recommendations is provided in Table 4.

Table 4

Summary of bulk carbide ceramic samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Silicon Carbide	○	✓	✓	-0.97	-0.05	0.00	130412C_A3	130425D_A6	130321G_A3
Tungsten Carbide	✓	✓	✓	0.00	0.00	-0.16	130723D_A1	130716E_A1	130709F_A2

Silicon Carbide (SiC) — Single-side polished, single crystal silicon carbide appeared to be stable in both NH₃ and NH₃-Cl environments, with the NH₃-Cl sample darkening slightly. Our results are in good agreement with gravimetric data in literature.¹² The NH₃-Na sample lost a small amount of mass without any observable change in thickness. The initially uniformly rough backside of the sample exhibited crystallographic features post-run, suggesting strong anisotropic etching. The sample appeared lighter in color, despite exhibiting more diffuse scattering of visible light from increased roughening of the originally unpolished surface. (See also Figure 17)

Figure 17 Representative silicon carbide samples: optical images (a) pre-run and post-run in (b) NH₃-Na (*possible uses*, ○), (c) NH₃ (*good to excellent potential*, ✓), or (d) NH₃-Cl (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Tungsten Carbide ((WC)₉₀₋₉₄Co₆₋₁₀Fe₀₋₄) — Tungsten carbide experienced no noticeable deterioration in its observed mechanical properties in any of the tested environments. The NH₃-Na sample experienced slight surface pitting. The NH₃-Cl sample lost a small amount of mass (35 mg), experienced an increase in diameter of +5 μm (+ 0.08 %), and exhibited patches of blue or brown coloration on the surface. The NH₃ sample experienced an increase in diameter of +4 μm (+ 0.07 %) without any significant weight loss. (See also Figure 18)

Figure 18 Representative tungsten carbide samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

3.2. Stability of Bulk Metals

3.2.1. Pure Metals and Metalloids

Summary — Pure metals and metalloids were investigated as potential high purity materials for use as construction materials as well as possible soluble agents. If an alloy of a pure metal was also studied then the pure metal will be discussed later with its alloys and will not appear in this section (cobalt, copper, iron, molybdenum, nickel, platinum, and tungsten). Pure metals that appeared to be chemically stable in the $\text{NH}_3\text{-Na}$ environment are silver and possibly niobium, tantalum, and vanadium, though of these only silver did not degrade mechanically due to hydrogen embrittlement. In the NH_3 environment gold, palladium, scandium, silicon, silver, titanium, and zirconium appeared to be stable. Gold, palladium, and vanadium appeared to be

stable in the $\text{NH}_3\text{-Cl}$ environment. Aluminum, germanium, magnesium, silicon, and yttrium appeared to be unstable and slightly soluble in the $\text{NH}_3\text{-Na}$ or $\text{NH}_3\text{-Cl}$ environment. A summary of the recommendations is provided in Table 5.

Table 5

Summary of pure material samples including recommendation (good to excellent potential ($\checkmark$), possible uses ($\bigcirc$), not recommended ($\times$)), relative weight change, and corresponding sample numbers. Metals for which their alloy system was also analyzed are excluded from this table.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$
Aluminum	$\times$		$\times$	-100.00		-100.00	130618C_A2		090512G_A1
Germanium	$\times$	$\bigcirc$	$\times$	N/A	-6.92	+42.33	130618C_A4	130702G_A3	130618E_A4
Gold	$\times$	$\checkmark$	$\checkmark$	-8.76	+0.13	+0.09	130716C_A1	130723G_A1	090524F_A1
Iridium	$\times$			-27.55			111027D_A1		
Lanthanum		$\times$			+77.00			090630C_A1	
Magnesium	$\times$		$\times$	-27.28		-100.00	130618C_A5		090606E_A1
Niobium	$\bigcirc$	$\bigcirc$	$\bigcirc$	+0.88	+0.71 -0.44	+0.89	090519B_A1	090630D_A1 130716G_A2	090429E_A1
Palladium	$\times$	$\checkmark$	$\checkmark$	-55.17	+0.21	+0.97	130716C_A2	130723G_A2	090524E_A1
Scandium		$\checkmark$			+3.18			090630B_A1	
Silicon	$\times$	$\checkmark$	$\times$	N/A	-0.07	+1.53	130618C_A3	130702G_A2	130618E_A3
Silver	$\checkmark$	$\checkmark$	$\times$	-0.04	0.00	-5.49	090519D_A1	130723G_A3	090429F_A1
Tantalum	$\bigcirc$	$\bigcirc$	$\times$	+0.32	-5.40	+1.17	090519C_A1	130716G_A3	090512F_A1
Titanium	$\times$	$\checkmark$	$\bigcirc$	+3.29 N/A	+0.20	+0.50	090611B_A1 130723C_A3	090530B_A1	130702E_A1
Vanadium	$\bigcirc$	$\bigcirc$	$\checkmark$	+1.45	+1.29	-0.42	130723C_A1	130716G_A1	090429G_A1
Yttrium	$\times$	$\times$	$\times$	-100.00	N/A	-100.00	090611C_A1	090530C_A1	130702E_A2

						-100.00			130723C_A2
Zirconium	×	✓	×	-0.28	+0.31	N/A	090611D_A1	090530D_A1	090512E_A1

Aluminum (Al) — Aluminum was not stable in the $\text{NH}_3\text{-Na}$ or $\text{NH}_3\text{-Cl}$ environment. It completely disintegrated and, in the case of $\text{NH}_3\text{-Na}$, formed white clumps. The residual material left in the autoclave after the $\text{NH}_3\text{-Cl}$ run was a mixture of powders with varying colors: gray-green, black and yellow-orange. There are numerous reports in literature on the solubility of aluminum in both ammonobasic^{21,22,23} and ammonoacidic^{24,25} conditions. AlN can also be formed if a temperature gradient is established^{22,23,25}, suggesting the possibility of forming a nitride layer on pure aluminum under certain conditions. The stability of aluminum in the NH_3 environment was not investigated. (See also Figure 19)

Figure 19 Representative aluminum samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ×) or (c) $\text{NH}_3\text{-Cl}$ (*not recommended*, ×). One square corresponds to 1 mm x 1 mm.

Germanium (Ge) — Germanium appeared to be unstable in all three environments. In the $\text{NH}_3\text{-Na}$ environment it cracked and exhibited white residue on its surface but still had a resolvable thickness decrease (-15 μm , -3 %). It is possible that the white residue is residual aluminum,

which was co-loaded with this sample. The sample exposed to $\text{NH}_3\text{-Cl}$ experienced a significant mass and thickness increase. It is interesting to note that literature would suggest germanium is soluble in $\text{NH}_3\text{-Cl}$ and does not appear to readily form a nitride.¹⁹ The weight increase of the germanium sample is therefore most likely a result of unintended interactions between co-loaded samples. The NH_3 sample turned very dark with a rough surface and exhibited measurable weight and thickness loss ($-28\text{ }\mu\text{m}$, -5%), suggesting it is soluble in the NH_3 solution. (See also Figure 20)

Figure 20 Representative germanium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*possible uses*, $\circ$), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to $1\text{ mm} \times 1\text{ mm}$.

Gold (Au) — Gold appeared to be stable in both NH_3 and $\text{NH}_3\text{-Cl}$ solutions. Neither sample exhibited any noticeable change in mechanical properties, most notably in malleability. These observations are in agreement with literature, wherein gold was used as a compliance layer in a multilayer coating of (Au | Pd | Pt) on Inconel 718.¹³ The $\text{NH}_3\text{-Na}$ sample blackened and sheets of material flaked off of the surface. It also experienced appreciable weight loss. These results are in slight contradiction to literature, wherein gold capsules may have been used under

ammonobasic conditions (although no analysis of gold's stability is provided).¹ (See also Figure 21)

Figure 21 Representative gold samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended, $\times$*), (c) NH_3 (*good to excellent potential, $\checkmark$*), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential, $\checkmark$*). One square corresponds to 1 mm x 1 mm.

Iridium (Ir) — Iridium was not chemically stable in the $\text{NH}_3\text{-Na}$ environment as evidenced by blackening of the surface and appreciable weight loss. The stability in NH_3 or $\text{NH}_3\text{-Cl}$ environments was not investigated. (See also Figure 22)

Figure 22 Representative iridium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

Lanthanum (La) — Lanthanum was not stable in the NH_3 environment. The material disintegrated and formed a white mass, which further disintegrated into a powder. Prior literature on the dissolution of lanthanum in $\text{NH}_3\text{-K}$ (350 °C, 400 MPa) would suggest the possible formation of $\text{La}(\text{NH}_2)_3$ ²⁶, while another reports suggests it is soluble in $\text{NH}_3\text{-Na}$ though not appreciably in NH_3 .^{27,28,29} Its stability in $\text{NH}_3\text{-Na}$ or $\text{NH}_3\text{-Cl}$ was not investigated. (See also Figure 23)

Figure 23 Optical image of post-run lanthanum sample after exposure to NH_3 (*not recommended*, $\times$). Diameter of powder containing glass beaker is 40 mm.

Magnesium (Mg) — The stability of magnesium was tested in the $\text{NH}_3\text{-Cl}$ and the $\text{NH}_3\text{-Na}$ environments. Complete disintegration occurred for the sample in $\text{NH}_3\text{-Cl}$ and after cleaning the autoclave with DI water no solid matter remained. This suggests that the sample either went into the supercritical solution during the run (and was purged from the autoclave while venting post-run) or was dissolved while neutralizing the autoclave post-run with DI water. Exposure to the $\text{NH}_3\text{-Na}$ environment caused the surface to roughen and the sample to become brittle and crack. Furthermore, the sample thinned uniformly ($\sim 806\ \mu\text{m}$, -76%) and dissolved without leaving white residue behind, suggesting the lost material either went into the supercritical solution during the run or was dissolved while neutralizing the autoclave post-run with DI water. Literature shows magnesium can form an amide in solution and that it is soluble in $\text{NH}_3\text{-Na}$ ^{30,31} or $\text{NH}_3\text{-K}$ ¹. (See also Figure 24)

Figure 24 Representative magnesium samples: optical image (a) pre-run and post-run in (b)

$\text{NH}_3\text{-Na}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

Niobium (Nb) — The niobium samples gained weight in all three environments. The H-Nb phase diagram would suggest that niobium has the ability to absorb appreciable amounts of hydrogen.³² Absorption of hydrogen into the lattice would cause the sample to expand, which is

consistent with the observed thickness increase of around +76 μm (+13 %). This volume change could be the origin of the sample becoming brittle and shattering in the NH_3 environment. Thus, in the NH_3 environment niobium is mechanically unstable. Investigation of the sample surfaces suggest that niobium is chemically stable in all three environments as no evidence of corrosion could be found. Literature would suggest though that niobium would have finite solubility in $\text{NH}_3\text{-Na}$ given the growth of LaNbON_2 from a La_2Nb alloy, though higher concentrations of oxygen were present when compared to our environment given their deliberate, minor addition of sodium hydroxide.³³ (See also Figure 25)

Figure 25 Representative niobium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (c) NH_3 (*possible uses*, $\bigcirc$), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, $\bigcirc$). One square corresponds to 1 mm x 1 mm.

Palladium (Pd) — Palladium was not stable in the $\text{NH}_3\text{-Na}$ environment. In addition to significant weight loss, the sample blackened and surface layers flaked off. The sample also became brittle and was easily chipped. In the NH_3 environment, palladium took on a matte, light gray surface finish. The black spot on the sample shown in Figure 26 (c) was most likely due to

unintended interactions with the co-loaded gold sample as the spot was located where the samples were touching. In the $\text{NH}_3\text{-Cl}$ environment a slight weight increase was observed along with a slight darkening of the surface. For both NH_3 and $\text{NH}_3\text{-Cl}$ samples, no changes in mechanical properties were observed. (See also Figure 26)

Figure 26 Representative palladium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Scandium (Sc) — Scandium appeared to be stable in the NH_3 environment. A slight weight increase was accompanied by visible yellow coloration of the surface of the sample pieces. (See also Figure 27)

Figure 27 Optical image of post-run scandium pieces after exposure to NH_3 (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Silicon (Si) — Single crystal silicon did not appear to be stable in either the $\text{NH}_3\text{-Na}$ or $\text{NH}_3\text{-Cl}$ environment. In the $\text{NH}_3\text{-Na}$ environment the sample broke apart and slightly dissolved, as may be anticipated given the formation of $\text{Si}_2\text{N}_2\text{NH}$ in $\text{NH}_3\text{-K}$ environments.³⁴ The white powder that was observed on the samples was most likely residual material from the co-loaded aluminum sample. In the $\text{NH}_3\text{-Cl}$ environment the sample appeared to have gained material on its surface, consistent with the overall weight increase and thickness change (+124 μm , +26 %). In the NH_3 environment the sample did not change appreciably and appeared to be stable. (See also Figure 28)

Figure 28 Representative silicon samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*good to excellent potential*, $\checkmark$), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

Silver (Ag) — Silver appeared to be stable in both the NH_3 and $\text{NH}_3\text{-Na}$ environments. No changes to its mechanical properties were observed. In the $\text{NH}_3\text{-Cl}$ environment the sample lost weight but remained malleable. The mass loss presumably occurred through the formation of silver chlorides. Literature reports that in $\text{NH}_3\text{-F}$ environments (addition of ammonium fluoride to pure ammonia) silver appears to be stable when used as a coating on Inconel 718¹³, so the chemical instability in $\text{NH}_3\text{-Cl}$ may not be a general feature of acidic supercritical ammonia solutions. A large number of runs have been performed in $\text{NH}_3\text{-Na}$ solutions with a silver capsule for the growth of GaN by the authors¹⁶ and a very small etch rate has been observed after >10,000 hrs exposure to $\text{NH}_3\text{-Na}$, suggesting silver has a very small but finite solubility in $\text{NH}_3\text{-Na}$ solutions. These observations are further substantiated by early success in using silver as a capsule under ammonobasic conditions.¹ (See also Figure 29)

Figure 29 Representative silver samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✗). One square corresponds to 1 mm x 1 mm.

Tantalum (Ta)—Tantalum did not appear to be stable in the $\text{NH}_3\text{-Cl}$ environment as evidenced by weight gain, surface color change, embrittlement, and cracking. The embrittlement of tantalum can be anticipated given reported hydride formation at higher temperatures from gaseous ammonia.³⁹ Literature reports mass loss for tantalum in $\text{NH}_3\text{-Cl}$ environments with distinct corrosion and complete dissolution in $\text{NH}_3\text{-F}$ environments (autoclave initially filled with ammonia and ammonium fluoride).¹³ In the NH_3 environment the sample decreased in weight while increasing in thickness (+17 μm , + 3 %). EDX analysis of the sample surface revealed high concentrations of O (~ 25 %) and N (~ 43 %) in addition to Ta (~ 29 %), suggesting the formation of tantalum nitride, tantalum oxide, and/or tantalum oxynitride (TaON). The surface formed a powder and was easily brushed off. When exposed to a $\text{NH}_3\text{-Na}$ environment, the sample turned dark and became brittle. It is interesting to note that the sample did not lose weight, as literature would suggest tantalum is soluble in $\text{NH}_3\text{-Na}$.²⁷ It is possible that a TaON surface layer formed, thereby slightly increasing its weight. (See also Figure 30)

Figure 30 Representative tantalum samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (c) NH_3 (*possible uses*, $\bigcirc$), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

Titanium (Ti) — Titanium gained weight in both the NH_3 and $\text{NH}_3\text{-Cl}$ environments. In NH_3 the sample surface turned a purple color, while it blackened in the $\text{NH}_3\text{-Cl}$ environment. No pitting or deterioration in mechanical properties was observed for either sample. A subsequent run in $\text{NH}_3\text{-Cl}$ using a capsule design showed signs of embrittlement. The capsule appeared to have self-sealed and then burst. The surfaces changed color to a yellow or dark green, though only mild surface corrosion was evident. Yellow dots formed on the interior surface of the capsule. Although signs of dissolution were not clearly evident, extreme nitridation was present. EDX analysis of the remaining material indicated titanium nitride formation (51 at% Ti, 49 at% N), though titanium hydride formation may also have occurred as hydrogen is not detectable using EDX. Two runs were performed in the $\text{NH}_3\text{-Na}$ environment with different outcomes. The sample which was co-loaded with vanadium, yttrium, and zirconium nitride-coated zirconium experienced complete disintegration, as did the co-loaded yttrium. A sample which was not co-loaded with any other material did not experience disintegration. Quite to the contrary, it slightly

gained mass and was covered by a gold-colored surface layer. This sample embrittled and cracked. Literature reports attempts of using TiN as a coating on Inconel 718, which resulted in the complete loss of the coating. This was most likely due to poor adhesion on the Inconel substrates.¹² (See also Figure 31)

Figure 31 Representative titanium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*good to excellent potential*, $\checkmark$), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, $\circ$). One square corresponds to 1 mm x 1 mm.

Vanadium (V) — The surface color of the vanadium samples from all three environments changed from a metallic grey to a rose-gold or brass color. In NH_3 the sample gained weight and saw an increase in thickness (+7 μm , +5 %). Subsequent EDX analysis revealed ~ 43 % V and ~ 48 % N, suggesting the formation of vanadium nitride. The sample also became brittle and cracked. In the $\text{NH}_3\text{-Na}$ environment the sample gained weight, despite an overall decrease in thickness by 10 μm (-7 %). The sample cracked upon removal, presumably due to embrittlement. For the $\text{NH}_3\text{-Cl}$ environment, the sample slightly lost mass but did not crack or appear to have

any modified mechanical properties. Interestingly, the roughened side of the sample turned black while the smooth surface turned a uniform rose-gold color. It is possible that vanadium nitride formed more rapidly on the rough surface and flaked off, resulting in the observed mass loss. (See also Figure 32)

Figure 32 Representative vanadium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (c) NH_3 (*possible uses*, $\bigcirc$), or $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, $\checkmark$) for an initially (d) smooth or (e) rough surface. One square corresponds to 1 mm x 1 mm.

Yttrium (Y) — Yttrium was not found to be stable in any of the environments. In all cases the samples completely disintegrated and formed a very fine, white powder. The white powder from the $\text{NH}_3\text{-Cl}$ and $\text{NH}_3\text{-Na}$ samples was soluble in water (these samples were co-loaded with other materials) but the powder from the NH_3 sample was not (this sample was not co-loaded with any other material). Literature reports the formation of $\text{Na}_3[\text{Y}(\text{NH}_2)_6]$ and $\text{Na}[\text{Y}(\text{NH}_2)_4]$ for $\text{NH}_3\text{-Na}$

at 250 °C and ~ 500 MPa ammonia, while $K_3Y(NH_2)_6$ was found for NH_3-K ³⁵. YN was found to form when starting from pure yttrium and NH_3 at 350 °C and 60 MPa.³⁶ (See also Figure 33)

Figure 33 Representative yttrium samples: optical images (a) pre-run and (b) remaining white powder contained in a plastic bag post-run in NH_3 (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

Zirconium (Zr) — In the NH_3-Cl environment zirconium was not stable and disintegrated, as evidenced by the residual fine black powder. In the NH_3-Na environment the sample became brittle and cracked into multiple pieces. The surface coloration ranged from light purple to dark yellow. In the NH_3 environment the sample appeared to be stable, without any observable changes in mechanical properties and a slight gain in weight. These observations compare well with literature, wherein it is reported that zirconium is stable up to 1000 °C when exposed to gaseous ammonia.³⁷ (See also Figure 34)

Figure 34 Representative zirconium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

3.2.2. Cobalt and Cobalt Alloys

Summary — Cobalt and its precipitate-hardened alloy with tungsten and aluminum (Co-W-Al) are very promising for their apparent corrosion resistance in the $\text{NH}_3\text{-Na}$ and NH_3 environments. The addition of W and Al appeared to stabilize the alloy in $\text{NH}_3\text{-Cl}$ as well, suggesting this material may be suitable for use as an alternative high temperature superalloy material for the walls or other wetted components of an autoclave system.³⁸ A summary of the recommendations is provided in Table 6.

Table 6

Summary of cobalt and cobalt alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Cobalt	✓	✓	×	+0.04	-0.01	-40.98	130604C_A1	130604G_A1	090606F_A1
						-41.54			130611F_A1
Co-W-Al Alloy	✓	✓	✓	-0.02	+0.07	-0.05	130604C_A2	130604G_A2	130611F_A2

Cobalt (Co) — Cobalt appeared to be stable in both NH₃ and NH₃-Na environments. Only minor weight change was observed, and post-run EDX analysis on the NH₃-Na sample suggested the surface was primarily composed of cobalt, oxygen, and a small amount of carbon. In the NH₃-Cl environment the cobalt was severely corroded and dissolved into solution. (See also Figure 35)

Figure 35 Representative cobalt samples: optical images (a) pre-run and post-run in (b) NH₃-Na (good to excellent potential, ✓), (c) NH₃ (good to excellent potential, ✓), or (d) NH₃-Cl (not recommended, ×). One square corresponds to 1 mm x 1 mm.

Cobalt-Tungsten-Aluminum Alloy ($\text{Co}_{80}\text{W}_{10.6}\text{Al}_{9.4}$) — The Co-W-Al alloy appeared to be stable in all three environments. Only small weight changes were observed in all the samples. Post-run EDX analysis on the NH_3 -Cl sample indicated lower concentrations of cobalt, tungsten, and aluminum on the surface as compared to their average bulk concentrations pre-run (80 % Co, 10.6 % W, 9.4 % Al). Decreased concentrations of the main alloying elements at the surface coupled with the presence of appreciable amounts of O and N indicates the formation of a surface oxide (possibly Al_2O_3 , given its observed stability in the NH_3 -Cl environment) and/or a surface nitride. The weight gain of the NH_3 sample was most likely due to the formation of a nitride, which is supported by EDX analysis. (See also Figure 36)

Figure 36 Representative cobalt-tungsten-aluminum samples: optical images (a) pre-run and post-run in (b) NH_3 -Na (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) NH_3 -Cl (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

3.2.3. Copper and Copper Alloys

Summary — Copper and its investigated alloys (Cu-Ni, Cu-Zn) were not stable in the $\text{NH}_3\text{-Cl}$ environment. In the NH_3 environment they appeared to be have corroded at a slower rate and, with the exception of the Cu-Ni alloy, also embrittled. These observations are in line with extensive literature on exposure for copper containing materials to gaseous or liquid ammonia.³⁹ In the $\text{NH}_3\text{-Na}$ environment only the Ni-Cu alloy appeared to be stable, though it gained weight. A summary of the recommendations is provided in Table 7.

Table 7

Summary of copper and copper alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$
Brass 260	×	○	×	-53.64	-9.29	-100.00	130521C_A3	130514G_A3	130425E_A3
Constantan (TC J-, TC E-)	○	✓	×	+2.00	-0.09	-100.00	130521C_A1	130514G_A1	130425E_A1
Copper	×	○	×	-100.00	-6.36	-100.00	130521C_A2	130514G_A2	130425E_A2

Copper (Cu) — Copper was not found to be stable in either the $\text{NH}_3\text{-Na}$ or $\text{NH}_3\text{-Cl}$ environment because of partial to complete disintegration or dissolution. The observed instability of copper under ammonobasic conditions is comparable to prior observations.¹ The $\text{NH}_3\text{-Cl}$ sample was co-loaded with other copper containing alloys which all fused into lumps, as seen in Figure 37 (d).

A study on copper's corrosion resistance in another acidic supercritical ammonia solution, $\text{NH}_3\text{-I}$ (autoclave was loaded with ammonia and ammonium iodide), similarly found that it was not stable.¹³ The copper sample also etched and became brittle in the NH_3 environment, though the weight loss was smaller (-18 mg, -6 %). Literature reports though that copper gaskets used during sealing of autoclaves are not attacked for supercritical NH_3 solutions.¹ The dark spots seen in Figure 37 (c) developed during cleaning of the sample post-run and were not present when removing from the autoclave. (See also Figure 37)

Figure 37 Representative copper samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*possible uses*, $\circ$), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

Brass 260 ($\text{Cu}_{71}\text{Zn}_{29}$) — Brass 260 was not stable in any of the three environments. The $\text{NH}_3\text{-Cl}$ sample fused with other co-loaded copper-containing samples. The NH_3 sample embrittled and thickened (+10 μm , +10 %) while losing mass (-53 mg, -9 %). Similarly to the pure copper sample, the Brass 260 sample developed black spots on its surface after exposure to water post-run. The $\text{NH}_3\text{-Na}$ sample did not completely dissolve but it lost more than half of its mass and 20 % of its thickness (-21 μm). The surface turned a dark brown color and the sample became embrittled. (See also Figure 38)

Figure 38 Representative brass 260 samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*possible uses*, $\circ$), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$).

One square corresponds to 1 mm x 1 mm.

Constantan (TC J-, TC E-) ($\text{Cu}_{53}\text{Ni}_{47}$) — The copper-nickel alloy Constantan was not stable in the $\text{NH}_3\text{-Cl}$ environment as it completely disintegrated and fused to the co-loaded copper-

containing samples. In the NH_3 and $\text{NH}_3\text{-Na}$ environments, the samples appeared to have been largely unaffected except for acquiring a dull, matte gray surface finish. The $\text{NH}_3\text{-Na}$ sample did gain a small amount of weight (+ 15.91 mg, +2 %) and parts of the surface appeared gold-colored while the rest of the surface turned a dull gray color. (See also Figure 39)

Figure 39 Representative constantan (TC J-, TC E-) samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (c) NH_3 (*good to excellent potential*, $\checkmark$), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

3.2.4. Iron and Iron Alloys

Summary — Iron and its alloys were not found to be stable in $\text{NH}_3\text{-Cl}$, though they fared better in the NH_3 and $\text{NH}_3\text{-Na}$ environments. All of the samples gained weight in NH_3 and $\text{NH}_3\text{-Na}$, and with the exception of the 1018 steel, did not exhibit any embrittlement post-run. EDX

analysis of the sample surfaces and polished cross-sections suggested the formation of a nitride surface layer, which is consistent with the weight gain and surface coloration. These observations are further substantiated by industry practices of considering iron and steels to be mostly stable when exposed to ammonia gas, while at higher temperatures ammonia dissociation may lead to the formation of a surface nitride layer.³⁹ A summary of the recommendations is provided in Table 8.

Table 8

Summary of iron and iron alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
1018 Steel	○	○	×	+2.58	+0.86	-66.95	130618C_A1	130702G_A1	130618E_A1
15-5 PH	○	○	×	+0.27	+0.88	-1.22	130514D_A1	130507E_A2	130416F_A2
17-4 PH	○	○	×	+0.25	+0.86	-1.96	130514D_A2	130507E_A3	130416F_A3
316L Stainless Steel	○	○	×	+0.81	+0.81	-0.08	130311D_A1	130507E_A1	130416F_A1
				+0.24			130311D_A2		
				+0.27	+2.46	+0.80	130311D_A3	130507E_A4	130416F_A4

1018 Steel (Fe_{98.3}Mn_{0.8}C_{0.9}) — Although 1018 steel was not catastrophically unstable, it exhibited neither mechanical nor chemical stability in the NH₃ and NH₃-Na environments. Both samples exhibited a white surface deposition, a small increase in thickness, a slight weight gain, and embrittlement. A few brown spots and spalling on the surface were observed for the NH₃

sample. The slight increase in weight can be attributed to the formation of an iron nitride or manganese nitride, which are known to form in supercritical ammonia solutions.^{40,41,42,43} Loss of Mn from the sample in NH₃-Na would be anticipated though as Na₂[Mn(NH₂)₄] is known to exist in NH₃-Na.⁴⁴ The weight increase of the NH₃-Na sample due to nitride formation may be masking loss of Mn. The NH₃-Cl sample was not stable and suffered from embrittlement, mass loss and, upon exposure to air and water, oxidation as evidenced by the rust-colored residual matter on the surface. The mass loss of this sample may be due to finite solubility of Fe in NH₃-Cl. Iron is known to be soluble in NH₃-I forming [Fe(NH₃)₆]I₂⁴⁵, while [Fe(NH₃)₆]Cl₂ was found to form 1 atm of ammonia from FeCl₂.⁴⁶ The embrittlement of the sample may be expected given the extensive literature on stress-corrosion cracking of steels in liquid ammonia and hydrogen attack at higher temperatures and pressures.³⁹ (See also Figure 40)

Figure 40 Representative 1018 steel samples: optical images (a) pre-run and post-run in (b) NH₃-Na (*possible uses*, ○), (c) NH₃ (*possible uses*, ○), or (d) NH₃-Cl (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

15-5 Precipitation-Hardened Stainless Steel (15-5 PH) (Fe_{75.9}Cr_{16.1}Ni_{4.5}Si_{2.0}Mn_{1.0}C_{0.3}Nb_{0.2}P_{0.1}

S_{0.1}) — 15-5 PH stainless steel appeared to be partially stable in the NH₃ and NH₃-Na environments. Both samples exhibited a slight weight gain which may be due to the formation of

a nitride layer at the surface. This conclusion was supported by EDX analysis and visual inspection of polished cross-sections of the samples. The $\text{NH}_3\text{-Cl}$ sample embrittled and appeared black after it was removed from the autoclave. The sample oxidized once it was exposed to water. (See also Figure 41)

Figure 41 Representative 15-5 PH samples: optical images post-run for (a) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (b) NH_3 (*possible uses*, $\bigcirc$), or (c) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

17-4 Precipitation-Hardened Stainless Steel (17-4 PH) ($\text{Fe}_{75.6}\text{Cr}_{17.1}\text{Ni}_{3.7}\text{Si}_{1.9}\text{Mn}_{1.0}\text{C}_{0.3}\text{Nb}_{0.2}\text{P}_{0.1}\text{S}_{0.1}$) — 17-4 PH stainless steel appeared to behave identically to the 15-5 PH samples in each of the three environments. (See also Figure 42)

Figure 42 Representative 17-4 PH samples: optical images post-run for (a) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (b) NH_3 (*possible uses*, $\bigcirc$), or (c) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

316L Stainless Steel ($\text{Fe}_{64.7}\text{Cr}_{18.1}\text{Ni}_{11.3}\text{Mn}_{2.0}\text{Si}_{2.0}\text{Mo}_{1.4}\text{C}_{0.4}\text{P}_{0.1}\text{S}_{0.1}$) — 316L stainless steel appeared to be partially stable in the NH_3 and $\text{NH}_3\text{-Na}$ environments, exhibiting slight weight gain. This was attributed to a nitride layer which was detected by EDX analysis. 316L did not appear to be stable in $\text{NH}_3\text{-Cl}$. All samples gained weight and experienced severe discoloration and moderate embrittlement. (See also Figure 43)

Figure 43 Representative 316L stainless steel samples: optical images post-run for (a) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (b) NH_3 (*possible uses*, $\bigcirc$), or (c) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

3.2.5. Molybdenum and Molybdenum Alloys

Summary — Molybdenum and its high strength TZM alloy (Ti-Zr-Mo) were found to be stable in all three environments, making this alloy system a promising candidate for universal use with any supercritical ammonia solutions and possibly negating the need for any corrosion resistant

liner or capsule system to achieve a high purity growth environment. Literature data suggest molybdenum is also stable in $\text{NH}_3\text{-F}$ environments (autoclave filled with ammonia and ammonium fluoride).¹³ In light of these results, a pure molybdenum capsule was used to grow GaN in a $\text{NH}_3\text{-Na}$ environment and has performed well without any significant degradation for extended periods of time. A summary of the recommendations is provided in Table 9.

Table 9

Summary of molybdenum and molybdenum alloy samples including recommendation (good to excellent potential ($\checkmark$), possible uses ($\circ$), not recommended ($\times$)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$	$\text{NH}_3\text{-Na}$	NH_3	$\text{NH}_3\text{-Cl}$
Molybdenum	$\checkmark$	$\checkmark$	$\checkmark$	+0.01	+0.03	0.00	130625D_A2	130625G_A2	090606G_A1
TZM	$\checkmark$	$\checkmark$	$\checkmark$	0.00	0.00	0.00	130625D_A1	130625G_A1	131101E_A1

Molybdenum (Mo) — Molybdenum appeared to be very stable in all three environments. Slight surface coloration was observed on all samples yielding dark or rainbow-colored surfaces (as seen on the $\text{NH}_3\text{-Cl}$ sample). These results are comparable to those in literature for molybdenum in $\text{NH}_3\text{-Cl}$ environments.¹³ No noticeable change in structural quality was observed. Subsequently, capsules were made which were identical in design to a prior, successful design made out of pure silver for the growth of GaN.¹⁶ Numerous growth runs of GaN in a $\text{NH}_3\text{-Na}$ environment were performed using pure molybdenum parts and no degradation has thus far been observed (> 2000 hrs) at temperatures below $\sim 500^\circ\text{C}$. Above this temperature, a small amount

of black powder formation on the exposed surfaces was observed. The quantity of powder decreased with increased usage and post-run cleaning of the same capsule and materials. (See also Figure 44)

Figure 44 Representative molybdenum samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Titanium-Zirconium-Molybdenum (TZM) ($\text{Mo}_{98.8}\text{Ti}_1\text{Zr}_{0.08}\text{C}_{0.16}$) — Similar to the pure molybdenum samples, these samples appeared to be stable in all three environments. No degradation in mechanical behavior was found post-run for any of the samples. Exposure to a $\text{NH}_3\text{-Cl}$ environment resulted in blue spots appearing on the surface of the rod, though no change in weight or diameter was observed. (See also Figure 45)

Figure 45 Representative titanium-zirconium-molybdenum (TZM) samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

3.2.6. Nickel and Nickel Alloys

Summary — Pure nickel and Hastelloy C-276 were found to be stable in NH_3 and $\text{NH}_3\text{-Na}$ environments. Exposed type K and type N thermocouples developed a dull grey to gold surface color in NH_3 and $\text{NH}_3\text{-Na}$, though they continued to function accurately for temperature measurements using the thermocouple junction. In $\text{NH}_3\text{-Cl}$ neither nickel nor any of its alloys were stable. Hastelloy C-276 appeared to be stable in unlined Inconel 625 autoclaves when co-loaded with other materials, though it was modestly etched when tested in a capsule. This could have been due to galvanic protection of the Hastelloy C-276 by less stable co-loaded materials, the reactor walls and/or the nickel gasket. A summary of the recommendations is provided in Table 10.

Table 10

Summary of nickel and nickel alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Chromel C (TC K+)									
/ AlumeI (TC K-)	○	○	×	+0.02	+0.36	-63.62	130528D_A2	130521G_A2	130514F_A1
Junction									
Hastelloy C-276	✓	✓	○	+0.02	+0.05	-0.01	130604C_A3	130604G_A3	130611F_A4
Nickel	✓	✓	×	+0.01	+0.05	-52.22	130528D_A1	130521G_A1	130611F_A3
Nicrosil (TC N+) /									
Nisil (TC N-)	○	○	×	+0.03	+0.28	-59.18	130528D_A3	130521G_A3	130514F_A2
Junction									

Nickel (Ni) — Nickel was not stable in the NH₃-Cl environment and etched severely. The surface of this sample appeared porous after the run. EDX analysis suggested the surface was primarily composed of nickel, carbon, and some oxygen. Chromium, chlorine, and nitrogen were found in significant amounts within the pores of the surface. In the NH₃ and NH₃-Na environments nickel appeared to be stable, without any significant change in structural quality, weight, or thickness, as could be anticipated from literature.³⁹ Analysis of these samples using EDX also suggested the formation of a nickel carbide layer, given the high concentrations of nickel, carbon and, to a lesser extent, oxygen that were observed. A lack of nitrogen on the surface suggests nickel nitride may not readily form, as compared to the formation of Ni₃N from

[Ni(NH₃)₆]Cl₂ in NH₃-Na at lower temperatures.⁴⁷ The carbide layer formation could explain the slight grey dulling of the originally shiny metallic surface. The source of carbon on the surface was unknown, though it was known to be present in the bulk at a concentration of 0.012 % and trace amounts of residual organic material on the autoclave walls cannot be ruled out. (See also Figure 46)

Figure 46 Representative nickel samples optical images (a) pre-run and post-run in (b) NH₃-Na (*good to excellent potential, ✓*), (c) NH₃ (*good to excellent potential, ✓*), or (d) NH₃-Cl (*not recommended, ✗*). One square corresponds to 1 mm x 1 mm.

Chromel C (TC K+) / Alumel (TC K-) Junction (Ni₈₈Cr₁₁) / (Ni₉₂Mn₂Al₄Si₂) — Both high nickel content leads of a type K thermocouple junction appeared to be stable in both NH₃ and NH₃-Na environments. A dulling of the surface finish along with coloring to a slight golden shade was observed along with a minor weight gain. Despite this, the junction still appeared to provide an accurate readout of the junction temperature when measured using a portable thermocouple reader. In the NH₃-Cl environment both leads were heavily corroded. The behavior

of Chromel C was found to behave similarly to the in-depth investigated alloy Inconel 718 ($\text{Ni}_{53}\text{Cr}_{19}\text{Fe}_{18}\text{Nb}_5\text{Mo}_3\text{Ti}_1\text{Al}_1$) in all three environments.⁹ (See also Figure 47)

Figure 47 Representative Chromel C (TC K+) / Alumel (TC K-) junction samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (c) NH_3 (*possible uses*, $\bigcirc$), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

Hastelloy C-276 ($\text{Ni}_{58.9}\text{Cr}_{18.7}\text{Mo}_{10.4}\text{Fe}_{6.2}\text{Co}_{2.7}\text{W}_{1.3}\text{Mn}_{1.1}\text{V}_{0.4}\text{Si}_{0.2}\text{P}_{0.1}\text{S}_{0.1}\text{C}_{0.1}$) — Hastelloy C-276 appeared fairly stable in both the NH_3 and $\text{NH}_3\text{-Na}$ environments and experienced only very slight weight gain and surface discoloration, as would be expected based on literature.³⁹ Little to no nitride coating was detectable in EDX analysis. Although Hastelloy C-276 lost weight in the $\text{NH}_3\text{-Cl}$ environment, the sample remained largely undamaged. The apparent resilience of this alloy to the $\text{NH}_3\text{-Cl}$ environment could be related to its well-known excellent resistance to attack by aqueous chloride solutions. Additionally, EDX analysis showed a thin nitride layer on the surface of the specimen and the surface of the specimen appeared blackened by exposure to the

NH₃-Cl environment. Although initial tests indicated this alloy was fairly stable, the subsequent capsule test revealed that it is resistant but not inert. The machined surfaces of the capsule had pitting that was especially pronounced within the capsule. The surface appeared to have a roughened surface with multiple small, highly reflective areas resulting in a sparkling appearance. Yellow and brown colorations were also present. (See also Figure 48)

Figure 48 Representative Hastelloy C-276 samples: optical images (a) pre-run and post-run in (b) NH₃-Na (*good to excellent potential*, ✓), (c) NH₃ (*good to excellent potential*, ✓), or (d) NH₃-Cl (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Nicrosil (TC N+) / Nisil (TC N-) Junction (Ni_{81.2}Cr_{15.7}Si_{2.8}Mg_{0.2}) / (Ni_{91.2}Si_{8.8}) — The type N thermocouple leads behaved comparably to type K leads. In NH₃ and NH₃-Na environments, both leads took on a dull grey to gold-colored appearance while slightly gaining weight. An accurate temperature readout was observed when connecting to a portable thermocouple reader. The NH₃-Cl sample was heavily corroded. (See also Figure 49)

Figure 49 Representative Nicrosil (TC N+) / Nisil (TC N-) junction samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, $\bigcirc$), (c) NH_3 (*possible uses*, $\bigcirc$), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, $\times$). One square corresponds to 1 mm x 1 mm.

3.2.7. Platinum and Platinum Alloys

Summary — Platinum and its alloys (Pt-Rh, Pt-Ru) appeared to be stable in both the NH_3 and $\text{NH}_3\text{-Cl}$ environments. Pure platinum disintegrated into a fine black powder in the $\text{NH}_3\text{-Na}$ environment. Most samples saw a slight increase in thickness. In the $\text{NH}_3\text{-Na}$ environment only the platinum-ruthenium alloy was found to be stable, whereas all the others were degraded and embrittled. Platinum-ruthenium appeared to be stable in all three environments suggesting its possible use as a universal, though expensive, liner material for supercritical ammonia solutions.

A summary of the recommendations is provided in Table 11.

Table 11

Summary of platinum and platinum alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Platinum (TC R-)	×	✓	✓	-100.00	+1.83	+1.63	130507C_A2	130507G_A3	130412E_A3
Platinum- Rhodium (TC R+)	×	✓	✓	-86.38	+1.70	+1.26	130507C_A3	130507G_A2	130412E_A2
Platinum- Ruthenium	✓	✓	✓	+2.27	+2.50	+5.06	130507C_A1	130507G_A1	130412E_A1

Platinum (TC R-) (Pt) — Platinum completely disintegrated into a fine black powder in the NH₃-Na environment, which was sufficiently catalytic to auto-ignite IPA in contact with air. Platinum was found to be stable in NH₃ and NH₃-Cl environments, in agreement with data in literature.¹¹ No change in mechanical properties was observed for the NH₃-Cl sample, though there was a slight darkening of the surface. The NH₃ sample increased slightly in weight and thickness (+8 μm, + 2 %). (See also Figure 50)

Figure 50 Representative platinum (TC R-) samples: optical images (a) pre-run and post-run in (b) NH_3 (*good to excellent potential*, ✓), or (c) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Platinum-Rhodium (TC R+) ($\text{Pt}_{78}\text{Rh}_{22}$) — The addition of rhodium to pure platinum slightly improved the chemical stability of the material in the $\text{NH}_3\text{-Na}$ environment, though appreciable weight loss still occurred and the sample embrittled and cracked. EDX analysis of the surface suggested a surface layer formed with low concentrations of rhodium. The outermost layer was found to have ~ 94 % Pt and ~ 4 % Rh, whereas inner layers were found to have ~ 74 % Pt and ~ 26 % Rh once the outer layer was removed. It was not possible to determine if the high Pt-containing outer layer was formed due to deposition from the co-loaded pure platinum sample. For both the NH_3 and $\text{NH}_3\text{-Cl}$ samples, the diameters increased noticeably (+8—14 μm , +2—4 %). A slight darkening of the surface of the $\text{NH}_3\text{-Cl}$ sample was observed, whereas the NH_3 sample retained its metallic luster. Both samples remained malleable. (See also Figure 51)

Figure 51 Representative platinum-rhodium (TC R+) samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Platinum-Ruthenium ($\text{Pt}_{91}\text{Ru}_9$) — In the NH_3 and $\text{NH}_3\text{-Cl}$ environments, platinum-ruthenium appeared to be stable. There was no degradation in mechanical properties but the samples did experience a slight increase in thickness. The $\text{NH}_3\text{-Cl}$ sample was initially covered in deposition upon removing it from the autoclave, but the deposition was easily cleaned-off with mild abrasives. Interestingly, the presence of ruthenium appeared to stabilize the material in the $\text{NH}_3\text{-Na}$ environment. While the foil slightly deformed during the run (presumably due to the thinness of the sample: 54 μm), it did not noticeably thin or lose any weight. Quite to the contrary, a slight weight gain was observed, though this could be due to the observed black deposits instead of uniform weight gain. The samples remained malleable and could potentially be used in all three environments. (See also Figure 52)

Figure 52 Representative platinum-ruthenium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

3.2.8. Tungsten and Tungsten Alloys

Summary — Tungsten and its investigated alloys (W-Ni-Cu, W-Re) were stable in all three environments. Slight discoloration of the surface occurred in $\text{NH}_3\text{-Cl}$ environments. A summary of the recommendations is provided in Table 12.

Table 12

Summary of tungsten and tungsten alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
High-Strength Durable Tungsten	✓	✓	○	-0.10	-0.01	-0.32	130611D_A2	130709G_A2	130625F_A2
Tungsten	✓	✓	○	-0.94	0.00	0.00 +0.05	130611D_A1	130709G_A1	090524G_A1 130625F_A1
Tungsten-Rhenium (TC C-)	✓	✓	✓	-0.02	-0.04	+0.10	130611D_A3	130709G_A3	130625F_A3
Tungsten-Rhenium (TC C+)	✓	✓	✓	-1.29	-0.04	+0.07	130611D_A4	130709G_A4	130625F_A4

Tungsten (W) — Tungsten appeared to be stable in all three environments with no observed change in mechanical properties. A slight darkening of the surface occurred for the NH₃ sample, while the NH₃-Cl sample exhibited some minor localized corrosion. Some of the samples appeared to have a flaky surface, which may have been deposition on the sample or be the tungsten itself. (See also Figure 53)

Figure 53 Representative tungsten samples: optical images (a) pre-run and post-run in (b) NH₃-Na (*good to excellent potential*, ✓), (c) in NH₃ (*good to excellent potential*, ✓), or (d) NH₃-Cl (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

High-Strength Durable Tungsten ($W_{74.7}Ni_{15.6}Cu_{9.6}$) — The addition of nickel and copper to tungsten did not noticeably change the corrosion resistance of the material. Samples from all three environments did not appear to have lost any significant mass. The change in surface coloration was more pronounced in the NH_3 -Cl environment, resulting in a golden hue with some isolated red and green spots. (See also Figure 54)

Figure 54 Representative high-strength durable tungsten samples: optical images (a) pre-run and post-run in (b) NH_3 -Na (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) NH_3 -Cl (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Tungsten-Rhenium (TC C-) ($W_{74}Re_{26}$) — This material appeared to be stable in all three environments, as no significant changes in mass, thickness, or mechanical properties were observed. A slight dulling and greying of the surfaces occurred. (See also Figure 55)

Figure 55 Representative tungsten-rhenium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Tungsten-Rhenium (TC C+) (W_{95}Re_5) — The lower rhenium content (5 %) tungsten-rhenium samples did not appear to behave any differently than those with a higher rhenium content (26 %). All samples appeared stable in all three environments. (See also Figure 56)

Figure 56 Representative tungsten-rhenium (TC C+) samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

4. Summary

In summary, 35 bulk metals, 2 bulk metalloids, and 17 bulk ceramics were exposed to three different supercritical ammonia solutions ($\text{NH}_3\text{-Na}$, NH_3 , $\text{NH}_3\text{-Cl}$) and analyzed for their chemical and mechanical stability in these solutions. This study was performed primarily to survey a large set of materials and identify a subset of materials for possible future, in-depth studies.

Almost all materials displayed some form of change in color or texture (roughening or dulling of a prior shiny surface). Notable exceptions included SiO_2 and sapphire which remained transparent and gold, silver, platinum-ruthenium ($\text{Pt}_{91}\text{Ru}_9$), and niobium which retained their metallic sheen in the $\text{NH}_3\text{-Cl}$ environment.

For the oxide ceramics, it was interesting to discover that zirconia was the only stable oxide in $\text{NH}_3\text{-Na}$. Most oxides were stable in NH_3 and those with a large number of different cations were more likely to display color changes. Approximately half the oxides were stable in $\text{NH}_3\text{-Cl}$. High alumina, silica and/or zirconia containing oxides were the most stable and magnesium oxide was the only oxide material that completely disintegrated. Our results are in agreement with published literature data, where available.

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4 Boron nitride did not fare well in any environment and was more heavily corroded in $\text{NH}_3\text{-Na}$
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6 than in NH_3 and $\text{NH}_3\text{-Cl}$. Silicon nitride was soluble in $\text{NH}_3\text{-Na}$, though it appeared to be stable
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8 in NH_3 and $\text{NH}_3\text{-Cl}$.
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11 The two carbide ceramics which were tested (silicon carbide and tungsten carbide) appeared to
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13 be stable in all three environments. A slight roughening of the silicon carbide in $\text{NH}_3\text{-Na}$
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15 suggested slight corrosion. The tungsten carbide samples slightly swelled in diameter by a few
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17 microns.
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22 Among the investigated noble metals only silver and a platinum-ruthenium alloy ($\text{Pt}_{91}\text{Ru}_9$) were
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24 found to be stable in $\text{NH}_3\text{-Na}$. All other samples disintegrated or appreciably reacted with the
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26 solution. All of the investigated noble metals appeared to be stable in NH_3 , while in $\text{NH}_3\text{-Cl}$ all
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28 except silver appeared to be chemically stable. Copper and its alloys were not stable in any
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30 solution with the sole exception of Constantan ($\text{Cu}_{53}\text{Ni}_{47}$) in pure NH_3 .
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35 Pure molybdenum, tungsten, and their respective alloys all fared exceptionally well in the three
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37 solutions suggesting they are suitable as universal materials from both a chemical and structural
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39 standpoint. Titanium, vanadium, niobium, and tantalum appeared to be chemically stable in all
40
41 three solutions, although they typically embrittled and most slightly gained weight. Cobalt and
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43 its cobalt-tungsten-aluminum alloy ($\text{Co}_{80}\text{W}_{10.6}\text{Al}_{9.4}$) appeared stable in all three solutions with
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45 the sole exception of pure cobalt in $\text{NH}_3\text{-Cl}$.
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50 Nickel was largely stable in $\text{NH}_3\text{-Na}$ and NH_3 and its alloys behaved comparably. They
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52 consistently took on a dull grey or golden surface color. Exposed thermocouples of type K and N
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54 remained functional post-run. In the $\text{NH}_3\text{-Cl}$ environment nickel and all of its alloys corroded.
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4 Iron and its alloys behaved comparably in $\text{NH}_3\text{-Na}$ and NH_3 and exhibited stability with minor
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6 weight gain. In $\text{NH}_3\text{-Cl}$, none of the samples were found to be stable.
7
8

9
10 Most other metals and metalloids investigated in this study were not stable in any of the three
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12 solutions and lost weight. Scandium appeared to be stable and slightly gained weight in pure
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14 NH_3 . Lanthanum completely reacted to form a white colored substance with a substantially
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16 higher weight while yttrium completely disintegrated in all three environments and formed a
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18 water soluble species for $\text{NH}_3\text{-Cl}$ and $\text{NH}_3\text{-Na}$ environments.
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25 26 **Acknowledgments** 27

28
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A. Appendix

A detailed summary of all the runs performed (Table A.1) and samples analyzed (Table A.2) for this study is provided. The run number is comprised of a letter which represents a specific autoclave preceded by a six digit number. Sample numbers are built on a run and add two alphanumeric digits to make each sample label unique. Samples with the same first seven alphanumeric digits were co-loaded in the run represented by those digits.

Table A.1

Summary of performed runs wherein specified amounts of ammonia, sodium, and ammonium chloride were placed in an autoclave made of either Inconel 625 (Inc625) or René 41 (R41). Duration at specified external wall temperature with resulting peak pressure is provided.

Run Nr.	Autoclave Material	Fill Amount (g)			Duration (days)	Peak Pressure (MPa)	T _{external} ^a (°C)
		NH ₃	Na	NH ₄ Cl			
090429E	Inc625	20.7		1.662	4	100	575
090429F	Inc625	20.7		1.618	4	96	575
090429G	Inc625	21.2		1.629	4	99	575
090512E	Inc625	20.7		1.622	4	102	575
090512F	Inc625	20.7		1.628	4	101	575
090512G	Inc625	20.8		1.620	4	99	575
090519B	R41	29.8	1.400		4	182	575
090519C	R41	29.9	1.400		4	178	575
090519D	R41	29.7	1.400		4	178	575

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2							
3							
4	090524E	Inc625	20.7		1.620	4	95 575
5							
6	090524F	Inc625	20.6		1.630	4	93 575
7							
8	090524G	Inc625	21.6		1.620	4	103 575
9							
10	090530B	R41	34.8			4	211 575
11							
12	090530C	R41	34.3			4	203 575
13							
14	090530D	R41	34.2			4	207 575
15							
16	090606E	Inc625	20.6		1.640	4	86 575
17							
18	090606F	Inc625	20.6		1.630	4	88 575
19							
20	090606G	Inc625	20.4		1.640	4	93 575
21							
22	090611B	R41	36.9	1.500		1*	236 575
23							
24	090611C	R41	37.1	1.500		4	232 575
25							
26	090611D	R41	36.9	1.500		4	237 575
27							
28	090630B	R41	38.2			4	246 565
29							
30	090630C	R41	38.4			3	256 565
31							
32	090630D	R41	38.4			4	243 565
33							
34	090713B	R41	36.5			6.25	243 575
35							
36	090713C	R41	36.1			6.25	234 575
37							
38	111027D	R41	22.80	1.500		5	174 575
39							
40	130222C	R41	26.57	1.765		6	241 575
41							
42	130222D	R41	26.56	1.770		6	218 575
43							
44	130311C	R41	26.86			6	232 575
45							
46	130311D	R41	26.77	1.800		6	250 575
47							
48	130312E	Inc625	16.06		1.605	6	105 575
49							
50	130321C	R41	20.49	1.800		6	249 525
51							
52	130321G	Inc625	14.42		1.500	6	97 575
53							
54	130412C	R41	21.26	1.414		6	208 545
55							
56	130412D	R41	21.34	1.401		8*	48 575
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58	130412E	Inc625	16.03		1.607	6	110 545
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130416D	R41	21.36	1.401		6	171	575
130416F	Inc625	14.47		1.450	6	112	550
130416G	Inc625	14.52		1.450	6	100	575
130425C	R41	23.56	1.553		6	239	575
130425D	R41	26.57			6	224	575
130425E	Inc625	14.43		1.450	6	101	575
130507C	R41	23.53	1.550		6	182	575
130507E	Inc625	11.48			6	87	575
130507G	Inc625	15.27			6	101	575
130514D	R41	25.04	1.646		6	252	575
130514F	Inc625	14.50		1.449	6	99	575
130514G	Inc625	15.24			6	102	575
130521C	R41	25.04	1.663		6	199	575
130521E	Inc625	14.43		1.452	6	109	575
130521G	Inc625	15.25			6	103	575
130528D	R41	25.05	1.641		6	218	575
130528F	Inc625	13.67		1.371	6	99	575
130528G	Inc625	14.49			6	98	575
130604C	R41	25.04	1.657		6	207	575
130604E	Inc625	13.74		1.371	6	91	575
130604G	Inc625	14.50			6	104	575
130611D	R41	25.80	1.709		6	210	575
130611F	Inc625	13.74		1.376	6	100	575
130611G	Inc625	14.50			6	101	575
130618C	R41	25.80	1.704		6	234	575
130618E	Inc625	13.75		1.375	6	80	575
130618G	Inc625	14.49			6	103	575
130625D	R41	25.81	1.702		6	213	575

130625F	Inc625	13.74	1.372	6	95	575
130625G	Inc625	14.49		6	100	575
130702C	R41	25.81	1.702	6	260	540
130702E	Inc625	13.75	1.370	6	91	575
130702G	Inc625	14.51		3	110	575
130709D	R41	25.81	1.700	6	232	575
130709F	Inc625	13.75	1.366	6	89	575
130709G	Inc625	14.49		6	100	575
130716C	R41	25.81	1.704	6	236	575
130716E	Inc625	14.51		6	104	575
130716G	Inc625	14.51		6	98	575
130723C	R41	25.81	1.698	6	259	500
130723D	R41	25.81	1.701	6	218	575
130723G	Inc625	14.48		6	100	575
131101E	Inc625	11.47	1.137	12	85	575

*) Indicate runs during which a leak occurred.

a) Internal fluid temperature is estimated to be ~80 °C cooler

Table A.2

Summary of samples and their corresponding run (first 7 alphanumeric digits of the sample number) investigated in this study

Sample Nr.	Environment*	Name*	Nominal Chemical Composition	Type	Source	Product*	Purity (%)	Form	Sample Size	Weight (g)	
									(L x W x H) or (DIA x L) (all in mm)	Before	After
090429E_A1	NH ₃ -Cl	Niobium	Nb	PC	Alfa Aesar	14358	99.8	Sheet	28 x 15 x 0.5	1.69636	1.71148
090429F_A1	NH ₃ -Cl	Silver	Ag	PC	Alfa Aesar	39181	99.9	Sheet	25 x 11 x 0.5	1.41843	1.34052
090429G_A1	NH ₃ -Cl	Vanadium	V	PC	Alfa Aesar	14633	99.5	Sheet	27 x 16 x 0.25	0.60827	0.6057
090512E_A1	NH ₃ -Cl	Zirconium	Zr	PC	Alfa Aesar	10591	99.8	Sheet	25 x 10 x 0.25	0.46185	0
090512F_A1	NH ₃ -Cl	Tantalum	Ta	PC	Alfa Aesar	10355	99.95 (ex. Nb)	Sheet	27 x 10 x 0.5	1.92269	1.94512
090512G_A1	NH ₃ -Cl	Aluminum	Al	PC	Alfa Aesar	40531	99.99	Sheet	25 x 10 x 0.5	0.42331	0
090519B_A1	NH ₃ -Na	Niobium	Nb	PC	Alfa Aesar	14358	99.8	Sheet	23 x 15 x 0.5	1.47592	1.48894
090519C_A1	NH ₃ -Na	Tantalum	Ta	PC	Alfa Aesar	10355	99.95 (ex. Nb)	Sheet	25 x 15 x 0.5	2.90061	2.90986
090519D_A1	NH ₃ -Na	Silver	Ag	PC	Alfa Aesar	39181	99.9	Sheet	25 x 14 x 0.5	1.75289	1.75224
090524E_A1	NH ₃ -Cl	Palladium	Pd	PC	Alfa Aesar	11514	99.9	Sheet	26 x 8 x 0.5	1.04162	1.05174
090524F_A1	NH ₃ -Cl	Gold	Au	PC	Alfa Aesar	36148	99.95	Sheet	27 x 9 x 0.5	2.07708	2.07902
090524G_A1	NH ₃ -Cl	Tungsten	W	PC	Alfa Aesar	10414	99.95	Sheet	25 x 12 x 0.5	2.64294	2.64291
090530B_A1	NH ₃	Titanium	Ti	PC	Alfa Aesar	43676	99.99+	Sheet	26 x 7 x 0.5	0.42967	0.43054

090530C_A1	NH ₃	Yttrium	Y	PC	Alfa Aesar	616	99.9	Sheet	24 x 6 x 0.64	0.42026	0
090530D_A1	NH ₃	Zirconium	Zr	PC	Alfa Aesar	10591	99.8	Sheet	24 x 17 x 0.25	0.6305	0.63243
090606E_A1	NH ₃ -Cl	Magnesium	Mg	PC	Alfa Aesar	40604	99.9	Sheet	24 x 8 x 1	0.37224	0
090606F_A1	NH ₃ -Cl	Cobalt	Co	PC	Alfa Aesar	42659	99.95	Sheet	27 x 9 x 0.5	1.061	0.62618
090606G_A1	NH ₃ -Cl	Molybdenum	Mo	PC	Alfa Aesar	10045	99.95	Sheet	27 x 10 x 0.5	1.39249	1.39251
090611B_A1	NH ₃ -Na	Titanium	Ti	PC	Alfa Aesar	43676	99.99+	Sheet	18 x 15 x 0.5	0.49011	0.50624
090611C_A1	NH ₃ -Na	Yttrium	Y	PC	Alfa Aesar	616	99.9	Sheet	17 x 11 x 0.64	0.56599	0
090611D_A1	NH ₃ -Na	Zirconium	Zr	PC	Alfa Aesar	10591	99.8	Sheet	17 x 12 x 0.25	0.60692	0.60524
090630B_A1	NH ₃	Scandium	Sc	PC	Alfa Aesar	39996	99.9	Pieces	between 1-5	2.103	2.1699
090630C_A1	NH ₃	Lanthanum	La	PC	Alfa Aesar	175	99.9	Pieces	between 1-8	4.166	7.37373
090630D_A1	NH ₃	Niobium	Nb	PC	Alfa Aesar	14358	99.8	Sheet	24 x 17 x 0.5	1.74535	1.75782
111027D_A1	NH ₃ -Na	Iridium	Ir	PC	Furuya Metal	1184269		Sheet	5 x 5 x 1	0.53691	0.38900
130222C_A1	NH ₃ -Na	Sapphire	Al ₂ O ₃	SC	Namiki	SSP		Sheet	10 x 10 x 0.4	0.18418	0.08873
130222C_A2		Sapphire	Al ₂ O ₃	SC	Namiki	DSP		Sheet	10 x 10 x 0.4	0.24157	0.12769
130222D_A1	NH ₃ -Na	Sintered Alumina	Al ₂ O ₃	PC	McDanel		99.8	Sheet	10 x 10 x 3	2.33888	2.2436
130311C_A1	NH ₃	Sapphire	Al ₂ O ₃	SC	Namiki	SSP		Sheet	12 x 6 x 0.4	0.12372	0.12372
130311C_A2		Sapphire	Al ₂ O ₃	SC	Namiki	DSP		Sheet	13 x 6 x 0.4	0.14239	0.14235
130311C_A3		Sintered Alumina	Al ₂ O ₃	PC	McDanel		99.8	Sheet	10 x 10 x 3	1.19212	1.19155
130311D_A1	NH ₃ -Na	316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	Swagelok			Tube	6.4 DIA x 100	12.14854	12.24657
130311D_A2		316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	OnlineMetals.co			Rod	6.4 DIA x 100	25.05155	25.11253

130311D_A3		316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	OnlineMetals.com		Rod	6.4 DIA x 100	24.89881	24.96595	
130312E_A1	NH ₃ -Cl	Sapphire	Al ₂ O ₃	SC	Namiki	SSP	Sheet	12 x 5 x 0.4	0.12322	0.12321	
130312E_A2		Sapphire	Al ₂ O ₃	SC	Namiki	DSP	Sheet	10 x 10 x 0.4	0.11996	0.11996	
130312E_A3		Sintered Alumina	Al ₂ O ₃	PC	McDanel	99.8	Sheet	10 x 10 x 3	1.95348	1.95273	
130321C_A1	NH ₃ -Na	Pyrolytic Boron Nitride	BN	PC	Momentive		Sheet	15 x 7 x 1	0.19078	0.15306	
130321C_A2		Hot Pressed Boron Nitride	BN	PC	Saint Gobain		Sheet	13x 8 x 1.5	0.3511	0.33177	
130321C_A3		Silicon Nitride	Si ₃ N ₄	PC	Bomas Machine Specialties		Rod	26 x 4 x 3	1.00132	0.98574	
130321G_A1	NH ₃ -Cl	Quartz	SiO ₂	SC	MTI Corp	DSP	99.99	Sheet	10 x 10 x 0.5	0.13116	0.13116
130321G_A2		Soda lime	Si _{24.4} Al _{0.5} K _{0.6} Mg _{2.3} Na _{10.0} Ca _{2.5} O _{59.7}	A	Goldseal	3010	Sheet	10 x 8 x 1	0.20036	0.1975	
130321G_A3		Silicon Carbide	SiC	SC	NovaSiC	SSP	Sheet	8 x 8 x 0.4	0.08593	0.08593	
130412C_A1	NH ₃ -Na	Quartz	SiO ₂	SC	MTI Corp	DSP	99.99	Sheet	10 x 10 x 0.5	0.13441	0
130412C_A2		Soda lime	Si _{24.4} Al _{0.5} K _{0.6} Mg _{2.3} Na _{10.0} Ca _{2.5} O _{59.7}	A	Goldseal	3010	Sheet	10 x 7 x 1	0.1862	0	
130412C_A3		Silicon Carbide	SiC	SC	NovaSiC	SSP	Sheet	9 x 7 x 0.4	0.0957	0.09477	
130412E_A1	NH ₃ -Cl	Platinum-Ruthenium	Pt ₉₁ Ru ₉	PC	Alfa Aesar	42686	Sheet	20 x 20 x 0.05	0.54379	0.57132	
130412E_A2		Rhodium (TC R+)	Pt ₇₈ Rh ₂₂	PC	Omega	P13R-015	Wire	0.4 DIA x 150	0.5608	0.56787	

130412E_A3	Platinum (TC R-)	Pt	PC	Omega	P13R-015	Wire	0.4 DIA x 64	0.6081	0.61804
Hafnium									
130416D_A1	Nitride coated	HfN / Al ₂ O ₃	C			Sheet	25 x 11 x 0.4	0.73843	0
	NH ₃ -Na	Sapphire							
130416D_A2	Silicon Nitride	Si ₃ N ₄	PC	Bomas Machine Specialties		Rod	23 x 4 x 3	0.96198	0.88822
130416F_A1	316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	OnlineMetals.co m		Rod	6.3 DIA x 100	24.74875	24.72780
130416F_A2	15-5 PH	Fe _{75.9} Cr _{16.1} Ni _{4.5} Si _{2.0} Mn _{1.0} C _{0.3} N _{0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.co m		Rod	6.3 DIA x 75	18.30252	18.07854
130416F_A3	17-4 PH	Fe _{75.6} Cr _{17.1} Ni _{3.7} Si _{1.9} Mn _{1.0} C _{0.3} N _{0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.co m		Rod	6.3 DIA x 72	17.68322	17.33642
130416F_A4	316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	Swagelok		Tube	6.3 DIA x 100	12.18927	12.28712
Hafnium									
130416G_A1	Nitride coated	HfN / Al ₂ O ₃	C			Sheet	30 x 13 x 0.4	0.85264	0.85249
	Sapphire								
130416G_A2	Silicon Nitride	Si ₃ N ₄	PC	Bomas Machine Specialties		Rod	23 x 4 x 3	0.9041	0.90389
130416G_A3	Pyrolytic Boron Nitride	BN	PC	Momentive		Sheet	13 x 7 x 1	0.15888	0.16021
130416G_A4	Hot Pressed Boron Nitride	BN	PC	Saint Gobain		Sheet	13x 10 x 1.5	0.42931	0.42843
130425C_A1	Magnesium	MgO	SC	MTI Corp	DSP	Sheet	10 x 10 x 0.5	0.18672	0.17916

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		Oxide								
		Yttria-								
130425C_A2		stabilized	Zr ₂₃ Y ₄ O ₅₂	SC	MTI Corp		Sheet	9 x 9 x 0.5	0.26417	0.26405
		Zirconia								
		Hot Pressed								
130425D_A1		Boron Nitride	BN	PC	Saint Gobain		Sheet	13 x 10 x 1.5	0.44452	0.44212
		Pyrolytic								
130425D_A2		Boron Nitride	BN	PC	Momentive		Sheet	15 x 7 x 1	0.17435	0.17165
		Silicon Nitride								
130425D_A3		Si ₃ N ₄		PC	Bomas Machine Specialties		Rod	23 x 4 x 3	0.94406	0.94388
	NH ₃	Hafnium								
130425D_A4		Nitride coated	HfN / Al ₂ O ₃	C			Sheet	30 x 7 x 0.4	0.27729	0.27726
		Sapphire								
		Titanium								
130425D_A5		Nitride coated	TiN / Al ₂ O ₃	C			Sheet	18 x 10 x 0.4	0.33621	0.33621
		Sapphire								
130425D_A6		Silicon Carbide	SiC	SC	NovaSiC	SSP	Sheet	9 x 9 x 0.4	0.10651	0.10646
		Constantan (TC								
130425E_A1		J-, TC E-)	Cu ₅₃ Ni ₄₇	PC	Omega	IRCO-015	Wire	0.4 DIA x 64	0.80144	0
	NH ₃ -Cl	Copper	Cu	PC	Alfa Aesar	36395 99.998	Sheet	24 x 24 x 0.5	0.28907	0
130425E_A3		Brass 260	Cu ₇₁ Zn ₂₉	PC	Alfa Aesar	45181	Sheet	25 x 22 x 0.5	0.54517	0
		Platinum-								
130507C_A1		Ruthenium	Pt ₉₁ Ru ₉	PC	Alfa Aesar	42686	Sheet	20 x 10 x 0.05	0.27841	0.28472
	NH ₃ -Na	Platinum (TC								
130507C_A2		R-)	Pt	PC	Omega	P13R-015	Wire	0.4 DIA x 38	0.27741	0

		Platinum-								
130507C_A3		Rhodium (TC R+)	Pt ₇₈ Rh ₂₂	PC	Omega	P13R-015	Wire	0.4 DIA x 51	0.34255	0.04666
130507E_A1		316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	OnlineMetals.co m		Rod	6.3 DIA x 100	24.79494	24.9951
130507E_A2		15-5 PH	Fe _{75.9} Cr _{16.1} Ni _{4.5} Si _{2.0} Mn _{1.0} C _{0.3} N _{b0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.co m		Rod	6.3 DIA x 73	17.81681	17.97319
130507E_A3	NH ₃	17-4 PH	Fe _{75.6} Cr _{17.1} Ni _{3.7} Si _{1.9} Mn _{1.0} C _{0.3} N _{b0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.co m		Rod	6.3 DIA x 73	17.91821	18.07289
130507E_A4		316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	Swagelok		Tube	6.3 DIA x 100	11.9654	12.26012
130507G_A1		Platinum- Ruthenium	Pt ₉₁ Ru ₉	PC	Alfa Aesar	42686	Sheet	20 x 10 x 0.05	0.25308	0.25941
130507G_A2	NH ₃	Platinum- Rhodium (TC R+)	Pt ₇₈ Rh ₂₂	PC	Omega	P13R-015	Wire	0.4 DIA x 51	0.55248	0.56187
130507G_A3		Platinum (TC R-)	Pt	PC	Omega	P13R-015	Wire	0.4 DIA x 76	0.60912	0.62025
130514D_A1		15-5 PH	Fe _{75.9} Cr _{16.1} Ni _{4.5} Si _{2.0} Mn _{1.0} C _{0.3} N _{b0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.co m		Rod	6.3 DIA x 74	18.13513	18.18329
130514D_A2	NH ₃ -Na	17-4 PH	Fe _{75.6} Cr _{17.1} Ni _{3.7} Si _{1.9} Mn _{1.0} C _{0.3} N _{b0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.co m		Rod	6.3 DIA x 74	18.20757	18.25328
130514F_A1	NH ₃ -Cl	Chromel C (TC K+) / Alumel (TC K-)	Ni ₈₉ Cr ₁₁ / Ni ₉₂ Mn ₂ Al ₄ Si ₂	J	Omega	KMQIN	Wire	0.5 DIA x 101	3.24486	1.18063

		Junction									
		Nicrosil (TC									
		N+) / Nisil (TC	Ni _{81.2} Cr _{15.7} Si _{2.8} Mg _{0.2} /	J	Omega	NMQXL		Wire	0.5 DIA x 101	3.52521	1.43892
		N-) Junction	Ni _{91.2} Si _{8.8}								
		Constantan (TC									
		J-, TC E-)	Cu ₅₃ Ni ₄₇	PC	Omega	IRCO-015		Wire	0.4 DIA x 64	0.76539	0.76472
	NH ₃	Copper	Cu	PC	Alfa Aesar	36395	99.998	Sheet	24 x 24 x 0.5	0.28804	0.26972
		Brass 260	Cu ₇₁ Zn ₂₉	PC	Alfa Aesar	45181		Sheet	25 x 22 x 0.5	0.56945	0.51656
		Constantan (TC									
		J-, TC E-)	Cu ₅₃ Ni ₄₇	PC	Omega	IRCO-015		Wire	0.4 DIA x 64	0.79359	0.8095
	NH ₃ -Na	Copper	Cu	PC	Alfa Aesar	36395	99.998	Sheet	24 x 24 x 0.5	0.20301	0
		Brass 260	Cu ₇₁ Zn ₂₉	PC	Alfa Aesar	45181		Sheet	25 x 22 x 0.5	0.51921	0.24072
		Alumina									
		Silicate	Al _{13.1} Si _{21.0} Fe _{0.8} K _{0.6} Ti _{0.4} O _{64.1}	PC	McMaster	8479K41		Sheet	31 x 16 x 6	7.71861	7.72682
	NH ₃ -Cl	Glass Mica	Si _{15.4} Mg _{8.5} F _{4.2} Al _{3.2} K _{2.1} B _{2.0} O _{64.5}	A	McMaster	8489K61		Sheet	30 x 15 x 7	7.33903	7.3069
		Fused Silica	SiO ₂	A	McMaster	1357T41	99.995	Sheet	12 DIA x 1.6	0.43824	0.43429
		Nickel	Ni	PC	Alfa Aesar	44824	99.5	Sheet	100 x 12 x 1	10.60294	10.60829
		Chromel C (TC									
		K+) / Alumel									
		(TC K-)	Ni ₈₉ Cr ₁₁ / Ni ₉₂ Mn ₂ Al ₄ Si ₂	J	Omega	KMQIN		Wire	0.5 DIA x 101	3.24927	3.26095
	NH ₃	Junction									
		Nicrosil (TC									
		N+) / Nisil (TC	Ni _{81.2} Cr _{15.7} Si _{2.8} Mg _{0.2} /	J	Omega	NMQXL		Wire	0.5 DIA x 101	3.61343	3.62358
		N-) Junction	Ni _{91.2} Si _{8.8}								
		Nickel	Ni	PC	Alfa Aesar	44824	99.5	Sheet	100 x 12 x 1	10.62452	10.62556

		Chromel C (TC									
		K+) / Alumel									
130528D_A2		(TC K-)	Ni ₈₉ Cr ₁₁ / Ni ₉₂ Mn ₂ Al ₄ Si ₂	J	Omega	KMQIN		Wire	0.5 DIA x 101	3.24131	3.24205
		Junction									
		Nicrosil (TC									
130528D_A3		N+) / Nisil (TC	Ni _{81.2} Cr _{15.7} Si _{2.8} Mg _{0.2} /	J	Omega	NMQXL		Wire	0.5 DIA x 101	3.53565	3.53678
		N-) Junction	Ni _{91.2} Si _{8.8}								
130528F_A1		Glass Ceramic	Si _{20.7} Al _{7.9} Li _{3.2} Mg _{1.9} Ca _{1.4} Na _{0.6}	A	McMaster	84815K49		Sheet	40 x 10 x 5	6.35095	6.34659
			Ti _{0.6} Zn _{0.6} Ba _{0.5} K _{0.4} Zr _{0.4} O _{61.8}								
		Machinable									
130528F_A2	NH ₃ -Cl	High Alumina	Al ₂ O ₃	PC	McMaster	8978T61	99	Sheet	38 x 8 x 1.5	1.32229	1.38315
		Ceramic									
130528F_A3		Zirconia	ZrO ₂	PC	McMaster	8750K37	97	Rod	6.3 DIA x 50	9.2357	9.22606
130528G_A1		Magnesium									
		Oxide	MgO	SC	MTI Corp	DSP		Sheet	10 x 10 x 0.5	0.1866	--
		Yttria-									
130528G_A2	NH ₃	stabilized	Zr ₂₃ Y ₄ O ₅₂	SC	MTI Corp			Sheet	10 x 10 x 0.5	0.27987	0.27977
		Zirconia									
130528G_A3		Soda lime	Si _{24.4} Al _{0.5} K _{0.6} Mg _{2.3} Na _{10.0} Ca _{2.5}	A	Goldseal	3010		Sheet	8 x 8 x 1	0.17734	--
			O _{59.7}								
130528G_A4		Quartz	SiO ₂	SC	MTI Corp	DSP	99.99	Sheet	10 x 8 x 0.5	0.10542	0.10528
130604C_A1		Cobalt	Co	PC	Alfa Aesar	42659	99.95	Sheet	14 x 10 x 0.5	0.66617	0.66642
130604C_A2	NH ₃ -Na	Co-W-Al Alloy	Co ₈₀ W _{10.6} Al _{9.4}	PC	UCSB			Ingot	14 x 14 x 1	1.90086	1.90048
130604C_A3		Hastelloy C-	Ni _{58.9} Cr _{18.7} Mo _{10.4} Fe _{6.2} Co _{2.7} W _{1.3}	PC	Fry Steel	Fry Alloy		Rod	6.3 DIA x 74	20.96741	20.97154
		276	Mn _{1.1} V _{0.4} Si _{0.2} P _{0.1} S _{0.1} C _{0.1}			C 276					

130604E_A1	NH ₃ -Cl	Magnesium Oxide	MgO	SC	MTI Corp	DSP		Sheet	10 x 10 x 0.5	0.17599	0
130604E_A2		Yttria- stabilized Zirconia	Zr ₂₃ Y ₄ O ₅₂	SC	MTI Corp			Sheet	10 x 9 x 0.5	0.28586	0.28501
130604G_A1	NH ₃	Cobalt	Co	PC	Alfa Aesar	42659	99.95	Sheet	18 x 7 x 0.5	0.55082	0.55079
130604G_A2		Co-W-Al Alloy	Co ₈₀ W _{10.6} Al _{9.4}	PC	UCSB			Ingot	9 x 9 x 0.8	0.7205	0.72104
130604G_A3		Hastelloy C- 276	Ni _{58.9} Cr _{18.7} Mo _{10.4} Fe _{6.2} Co _{2.7} W _{1.3} Mn _{1.1} V _{0.4} Si _{0.2} P _{0.1} S _{0.1} C _{0.1}	PC	Fry Steel	Fry Alloy C 276		Rod	6.3 DIA x 74	20.55137	20.56195
130611D_A1	NH ₃ -Na	Tungsten High-Strength Durable	W W _{74.7} Ni _{15.6} Cu _{9.6}	PC	Alfa Aesar	10414	99.95	Sheet	48 x 17 x 0.5	4.31817	4.27769
130611D_A2		Tungsten Tungsten- Rhenium (TC C-)	W ₇₄ Re ₂₆	PC	Omega	T5R-010		Wire	0.25 DIA x 80	0.32771	0.32764
130611D_A3		Tungsten- Rhenium (TC C+)	W ₉₅ Re ₅	PC	Omega	T5R-010		Wire	0.25 DIA x 110	0.32562	0.32143
130611F_A1		Cobalt	Co	PC	Alfa Aesar	42659	99.95	Sheet	18 x 10 x 0.5	0.71897	0.42031
130611F_A2		Co-W-Al Alloy	Co ₈₀ W _{10.6} Al _{9.4}	PC	UCSB			Ingot	14 x 14 x 1.6	3.32139	3.3196
130611F_A3	NH ₃ -Cl	Nickel	Ni	PC	Alfa Aesar	44824	99.5	Sheet	101 x 12 x 1	10.65129	5.08878
130611F_A4		Hastelloy C- 276	Ni _{58.9} Cr _{18.7} Mo _{10.4} Fe _{6.2} Co _{2.7} W _{1.3} Mn _{1.1} V _{0.4} Si _{0.2} P _{0.1} S _{0.1} C _{0.1}	PC	Fry Steel	Fry Alloy C 276		Rod	6.3 DIA x 85	23.60138	23.59985
130611G_A1	NH ₃	Glass Ceramic	Si _{20.7} Al _{7.9} Li _{3.2} Mg _{1.9} Ca _{1.4} Na _{0.6}	A	McMaster	84815K49		Sheet	50 x 10 x 5	6.36584	6.36729

			$\text{Ti}_{0.6}\text{Zn}_{0.6}\text{Ba}_{0.5}\text{K}_{0.4}\text{Zr}_{0.4}\text{O}_{61.8}$								
		Machinable									
130611G_A2		High Alumina	Al_2O_3	PC	McMaster	8978T61	99	Sheet	48 x 9 x 1.5	1.3535	1.34144
		Ceramic									
130611G_A3		Zirconia	ZrO_2	PC	McMaster	8750K37	97	Rod	6.3 DIA x 48	9.11963	9.1197
130618C_A1		1018 Steel	$\text{Fe}_{98.3}\text{Mn}_{0.8}\text{C}_{0.9}$	PC	Unknown			Sheet	19 x 13 x 0.9	3.39116	3.47881
130618C_A2		Aluminum	Al	PC	Alfa Aesar	40531	99.99	Sheet	23 x 12 x 0.5	0.13663	0
130618C_A3	NH ₃ -Na	Silicon	Si	SC	MTI Corp			Sheet	47 x 16 x 0.5	0.86671	--
130618C_A4		Germanium	Ge	SC	MTI Corp			Sheet	48 x 15 x 0.5	2.10573	--
130618C_A5		Magnesium	Mg	PC	Alfa Aesar	40604	99.9	Sheet	16 x 14 x 1	0.41244	0.29993
130618E_A1		1018 Steel	$\text{Fe}_{98.3}\text{Mn}_{0.8}\text{C}_{0.9}$	PC	Unknown			Sheet	20 x 13 x 0.9	3.38799	1.11986
130618E_A2		Aluminum	Al	PC	Alfa Aesar	40531	99.99	Sheet	23 x 12 x 0.5	0.12983	0
130618E_A3	NH ₃ -Cl	Silicon	Si	SC	MTI Corp			Sheet	30 x 18 x 0.5	0.69793	0.70861
130618E_A4		Germanium	Ge	SC	MTI Corp			Sheet	30 x 15 x 0.5	1.59984	2.27709
130618G_A1		Aluminum		PC	McMaster	8479K41		Sheet	30 x 15 x 6	7.67459	7.63696
	NH ₃	Silicate	$\text{Al}_{13.1}\text{Si}_{21.0}\text{Fe}_{0.8}\text{K}_{0.6}\text{Ti}_{0.4}\text{O}_{64.1}$								
130618G_A2		Glass Mica	$\text{Si}_{15.4}\text{Mg}_{8.5}\text{F}_{4.2}\text{Al}_{3.2}\text{K}_{2.1}\text{B}_{2.0}\text{O}_{64.5}$	A	McMaster	8489K61		Sheet	30 x 15 x 6	7.11173	7.11556
130618G_A3		Fused Silica	SiO_2	A	McMaster	1357T41	99.995	Sheet	12 DIA x 1.6	0.43108	0.43099
		Titanium-									
130625D_A1		Zirconium-		PC	Plansee			Rod	6.3 DIA x 67	16.07167	16.07175
	NH ₃ -Na	Molybdenum	$\text{Mo}_{99.6}\text{Ti}_{0.3}\text{Zr}_{0.1}$								
		(TZM)									
130625D_A2		Molybdenum	Mo	PC	Alfa Aesar	10045	99.95	Sheet	23 x 22 x 0.5	1.5448	1.54491
130625F_A1	NH ₃ -Cl	Tungsten	W	PC	Alfa Aesar	10414	99.95	Sheet	33 x 16 x 0.5	2.87509	2.87643

		High-Strength									
130625F_A2		Durable	W _{74.7} Ni _{15.6} Cu _{9.6}	PC	McMaster	8279k15		Rod	6.3 DIA x 49	29.59689	29.502
		Tungsten									
		Tungsten-									
130625F_A3		Rhenium (TC	W ₇₄ Re ₂₆	PC	Omega	T5R-010		Wire	0.25 DIA x 80	0.32017	0.3205
		C-)									
		Tungsten-									
130625F_A4		Rhenium (TC	W ₉₅ Re ₅	PC	Omega	T5R-010		Wire	0.25 DIA x 110	0.31876	0.31898
		C+)									
		Titanium-									
130625G_A1	NH ₃	Zirconium-									
		Molybdenum	Mo _{99.6} Ti _{0.3} Zr _{0.1}	PC	Plansee			Rod	6.3 DIA x 68	16.12363	16.12395
		(TZM)									
130625G_A2		Molybdenum	Mo	PC	Alfa Aesar	10045	99.95	Sheet	23 x 23 x 0.5	1.59113	1.59165
		Aluminum									
130702C_A1		Silicate	Al _{13.1} Si _{21.0} Fe _{0.8} K _{0.6} Ti _{0.4} O _{64.1}	PC	McMaster	8479K41		Sheet	30 x 13 x 8	7.5337	7.41185
	NH ₃ -Na										
130702C_A2		Glass Mica	Si _{15.4} Mg _{8.5} F _{4.2} Al _{3.2} K _{2.1} B _{2.0} O _{64.5}	A	McMaster	8489K61		Sheet	30 x 15 x 6	7.33845	7.00029
130702C_A3		Fused Silica	SiO ₂	A	McMaster	1357T41	99.995	Sheet	12 DIA x 1.6	0.43215	0
130702E_A1		Titanium	Ti	PC	Alfa Aesar	43676	99.99+	Sheet	15 x 12 x 0.5	0.38914	0.39107
	NH ₃ -Cl										
130702E_A2		Yttrium	Y	PC	Alfa Aesar	616	99.9	Sheet	15 x 14 x 0.6	0.60065	0
130702G_A1		1018 Steel	Fe _{98.3} Mn _{0.8} C _{0.9}	PC	Unknown			Sheet	39 x 13 x 0.9	3.50269	3.53286
130702G_A2	NH ₃	Silicon	Si	SC	MTI Corp			Sheet	30 x 18 x 0.5	0.65774	0.65729
130702G_A3		Germanium	Ge	SC	MTI Corp			Sheet	35 x 13 x 0.5	1.72135	1.60227
130709D_A1	NH ₃ -Na	Glass Ceramic	Si _{20.7} Al _{7.9} Li _{3.2} Mg _{1.9} Ca _{1.4} Na _{0.6} Ti _{0.6} Zn _{0.6} Ba _{0.5} K _{0.4} Zr _{0.4} O _{61.8}	A	McMaster	84815K49		Sheet	50 x 10 x 5	6.57817	5.15855

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		Machinable									
130709D_A2		High Alumina	Al ₂ O ₃	PC	McMaster	8978T61	99	Sheet	50 x 12 x 1.5	1.67698	0.12533
		Ceramic									
130709D_A3		Zirconia	ZrO ₂	PC	McMaster	8750K37	97	Rod	6.3 DIA x 48	9.25769	9.25703
		Zirconium									
130709F_A1	NH ₃ -Cl	Nitride coated	ZrN / Zr	C				Sheet	50 x 15 x 0.3	1.45324	0.05896
		Zirconium									
130709F_A2		Tungsten Carbide	(WC) ₉₀₋₉₄ Co ₆₋₁₀ Fe ₀₋₄	PC	McMaster	8788A163		Rod	6.3 DIA x 50	22.69704	22.66184
130709G_A1		Tungsten	W	PC	Alfa Aesar	10414	99.95	Sheet	32 x 17 x 0.5	3.26742	3.26734
		High-Strength									
130709G_A2		Durable	W _{74.7} Ni _{15.6} Cu _{9.6}	PC	McMaster	8279k15		Rod	6.3 DIA x 48	28.79129	28.7892
		Tungsten									
	NH ₃	Tungsten-									
130709G_A3		Rhenium (TC C-)	W ₇₄ Re ₂₆	PC	Omega	T5R-010		Wire	0.25 DIA x 80	0.32317	0.32304
		Tungsten-									
130709G_A4		Rhenium (TC C+)	W ₉₅ Re ₅	PC	Omega	T5R-010		Wire	0.25 DIA x 110	0.31836	0.31824
130716C_A1	NH ₃ -Na	Gold	Au	PC	Alfa Aesar	36148	99.95	Sheet	15 x 8 x 0.5	1.17406	1.07121
130716C_A2		Palladium	Pd	PC	Alfa Aesar	11514	99.9	Sheet	15 x 12 x 0.5	1.2353	0.55373
130716E_A1	NH ₃	Tungsten Carbide	(WC) ₉₀₋₉₄ Co ₆₋₁₀ Fe ₀₋₄	PC	McMaster	8788A163		Rod	6.3 DIA x 45	22.98623	22.98646
		Zirconium									
130716E_A2		Nitride coated	ZrN / Zr	C					Sheet	50 x 19 x 0.3	1.56085

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		Zirconium									
130716G_A1		Vanadium	V	PC	Alfa Aesar	14633	99.5	Sheet	36 x 18 x 0.14	0.54602	0.55307
130716G_A2	NH ₃	Niobium	Nb	PC	Alfa Aesar	14358	99.8	Sheet	46 x 17 x 0.6	3.85528	3.83817
130716G_A3		Tantalum	Ta	PC	Alfa Aesar	10355	99.95 (ex. Nb)	Sheet	30 x 23 x 0.5	6.81818	6.44971
130723C_A1		Vanadium	V	PC	Alfa Aesar	14633	99.5	Sheet	36 x 18 x 0.14	0.55322	0.56126
130723C_A2		Yttrium	Y	PC	Alfa Aesar	616	99.9	Sheet	24 x 13 x 0.7	0.88641	0
130723C_A3	NH ₃ -Na	Titanium	Ti	PC	Alfa Aesar	43676	99.99+	Sheet	20 x 14 x 0.3	0.3666	--
		Zirconium									
130723C_A4		Nitride coated	ZrN / Zr	C				Sheet	19 x 14 x 0.3	0.50199	0.03198
		Zirconium									
130723D_A1		Tungsten		PC	McMaster	8788A163		Rod	6.3 DIA x 48	22.99298	22.99294
		Carbide	(WC) ₉₀₋₉₄ Co ₆₋₁₀ Fe ₀₋₄								
	NH ₃ -Na	Zirconium									
130723D_A2		Nitride coated	ZrN / Zr	C				Sheet	30 x 17 x 0.3	1.56317	1.59069
		Zirconium									
130723G_A1		Gold	Au	PC	Alfa Aesar	36148	99.95	Sheet	14 x 7 x 0.5	0.8215	0.82257
130723G_A2	NH ₃	Palladium	Pd	PC	Alfa Aesar	11514	99.9	Sheet	16 x 11 x 0.5	1.23286	1.23547
130723G_A3		Silver	Ag	PC	EPSI Metals	Knd3023	99.99	Rod	18 x 10 x 9	16.73662	16.73661
		Titanium-									
131101E_A1	NH ₃ -Cl	Zirconium-		PC	Plansee			Rod	6.3 DIA x 38	6.83492	6.83523
		Molybdenum	Mo _{99.6} Ti _{0.3} Zr _{0.1}								
		(TZM)									

* Abbreviations: Polycrystalline (PC), Single Crystal (SC), Coating (C), Junction (J), Amorphous (A), Single Side Polished (SSP), Double Side Polished (DSP), Thermocouple wire (TC) from indicated type and lead (+/-)

* Environments: Autoclave filled with samples(s) and ammonia (NH_3), ammonia and sodium metal ($\text{NH}_3\text{-Na}$), or ammonia and ammonium chloride ($\text{NH}_3\text{-Cl}$)

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Figure Captions

Figure 1 Schematic of the experimental setup using a Ni-Cr superalloy autoclave (11) sealed using a nickel gasket (12), a needle valve made of 316 stainless steel (SS 316) (13), a rupture disc made of SS 316 (14), and a pressure transducer which is heated externally using ceramic insulated resistive heaters (21). Copper heat spreaders (22), centering rings (23) and thermal insulation (24) are used to improve temperature uniformity which is measured on the external surface of the autoclave using copper tipped (25) type K thermocouples (42). Samples (31) are situated at the bottom of the one inch inner diameter autoclave.

Figure 2 Representative aluminum silicate samples: optical images (a) pre-run, and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*possible uses*, $\circ$), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, $\checkmark$). One square corresponds to 1 mm x 1 mm.

Figure 3 Representative fused silica samples: optical images (a) pre-run and post-run in (b) NH_3 (*good to excellent potential*, $\checkmark$) or (c) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, $\checkmark$). One square corresponds to 1 mm x 1 mm.

Figure 4 Representative glass ceramic samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*good to excellent potential*, $\checkmark$), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, $\checkmark$). One square corresponds to 1 mm x 1 mm.

Figure 5 Representative glass mica samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, $\times$), (c) NH_3 (*possible uses*, $\circ$), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, $\circ$). One square corresponds to 1 mm x 1 mm.

Figure 6 Representative machinable high alumina ceramic samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 7 Representative magnesium oxide samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕) or (c) NH_3 (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 8 Representative quartz samples: optical images (a) pre-run and post-run in (b) NH_3 (*good to excellent potential*, ✓) or (c) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 9 Representative sapphire samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 10 Representative sintered alumina samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 11 Representative soda lime samples: optical images (a) pre-run and post-run in (b) NH_3 (*possible uses*, ○) or (c) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 12 Representative yttria-stabilized-zirconia samples: optical images (a) pre-run, and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 13 Representative zirconia samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 14 Representative hot-pressed boron nitride samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 15 Representative pyrolytic boron nitride samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 16 Representative silicon nitride samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 17 Representative silicon carbide samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 18 Representative tungsten carbide samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 19 Representative aluminum samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕) or (c) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 20 Representative germanium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 21 Representative gold samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 22 Representative iridium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 23 Optical image of post-run lanthanum sample after exposure to NH_3 (*not recommended*, ✕). Diameter of powder containing glass beaker is 40 mm.

Figure 24 Representative magnesium samples: optical image (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 25 Representative niobium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 26 Representative palladium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 27 Optical image of post-run scandium pieces after exposure to NH_3 (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 28 Representative silicon samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 29 Representative silver samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 30 Representative tantalum samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 31 Representative titanium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 32 Representative vanadium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (c) NH_3 (*possible uses*, ○), or $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓) for an initially (d) smooth or (e) rough surface. One square corresponds to 1 mm x 1 mm.

Figure 33 Representative yttrium samples: optical images (a) pre-run and (b) remaining white powder contained in a plastic bag post-run in NH_3 (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 34 Representative zirconium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 35 Representative cobalt samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 36 Representative cobalt-tungsten-aluminum samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 37 Representative copper samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 38 Representative brass 260 samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 39 Representative constantan (TC J-, TC E-) samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 40 Representative 1018 steel samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 41 Representative 15-5 PH samples: optical images post-run for (a) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (b) NH_3 (*possible uses*, ○), or (c) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 42 Representative 17-4 PH samples: optical images post-run for (a) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (b) NH_3 (*possible uses*, ○), or (c) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 43 Representative 316L stainless steel samples: optical images post-run for (a) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (b) NH_3 (*possible uses*, ○), or (c) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 44 Representative molybdenum samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 45 Representative titanium-zirconium-molybdenum (TZM) samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 46 Representative nickel samples optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 47 Representative Chromel C (TC K+) / Alumel (TC K-) junction samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 48 Representative Hastelloy C-276 samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 49 Representative Nicrosil (TC N+) / Nisil (TC N-) junction samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*possible uses*, ○), (c) NH_3 (*possible uses*, ○), or (d) $\text{NH}_3\text{-Cl}$ (*not recommended*, ✕). One square corresponds to 1 mm x 1 mm.

Figure 50 Representative platinum (TC R-) samples: optical images (a) pre-run and post-run in (b) NH_3 (*good to excellent potential*, ✓), or (c) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 51 Representative platinum-rhodium (TC R+) samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*not recommended*, ✕), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 52 Representative platinum-ruthenium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 53 Representative tungsten samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) in NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 54 Representative high-strength durable tungsten samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*possible uses*, ○). One square corresponds to 1 mm x 1 mm.

Figure 55 Representative tungsten-rhenium samples: optical images (a) pre-run and post-run in (b) $\text{NH}_3\text{-Na}$ (*good to excellent potential*, ✓), (c) NH_3 (*good to excellent potential*, ✓), or (d) $\text{NH}_3\text{-Cl}$ (*good to excellent potential*, ✓). One square corresponds to 1 mm x 1 mm.

Figure 56 Representative tungsten-rhenium (TC C+) samples: optical images (a) pre-run and post-run in (b) NH₃-Na, (c) NH₃, or (d) NH₃-Cl. One square corresponds to 1 mm x 1 mm.

Table 1

Summary of materials surveyed for their stability in supercritical ammonia solutions. Detailed information on the materials, including their nominal chemical composition, can be found in Table A.2.

Class	Subclass	Common Material Name*
Bulk Ceramic	Oxide	Aluminum Silicate, Fused Silica, Glass Ceramic, Glass
		Mica, Machinable High Alumina Ceramic, Magnesium Oxide, Quartz, Sapphire, Sintered Alumina, Soda lime,
		Ytria-stabilized Zirconia, Zirconia
	Nitride	Hot Pressed Boron Nitride, Pyrolytic Boron Nitride,
		Silicon Nitride
	Carbide	Silicon Carbide, Tungsten Carbide
Bulk Metal	Pure	Aluminum, Cobalt, Copper, Germanium, Gold, Iridium,
		Lanthanum, Magnesium, Molybdenum, Nickel, Niobium,
		Palladium, Platinum (TC R-), Scandium, Silicon, Silver,
		Tantalum, Titanium, Tungsten, Vanadium, Yttrium,
		Zirconium
	Cobalt Alloys	Cobalt-Tungsten-Aluminum Alloy
	Copper Alloys	Brass 260, Constantan (TC J-, TC E-)
	Iron Alloys	1018 Steel, 15-5 PH, 17-4 PH, 316L Stainless Steel
	Molybdenum Alloys	Titanium-Zirconium-Molybdenum
	Nickel Alloys	Chromel C (TC K+), Alumel (TC K-), Hastelloy C-276,

Nicrosil (TC N+), Nisil (TC N-)	
Platinum Alloys	Platinum-Rhodium (TC R+), Platinum-Ruthenium
Tungsten Alloys	High-Strength Durable Tungsten, Tungsten-Rhenium (TC C-), Tungsten-Rhenium (TC C+)

* Abbreviations: TC = Thermocouple, +/- indicates positive or negative lead from indicated thermocouple type.

Table 2

Summary of bulk oxide ceramic samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Aluminum Silicate	×	○	✓	-1.62	-0.49	+0.11	130702C_A1	130618G_A1	130521E_A1
Fused Silica	×	✓	✓	-100.00	-0.02	-0.90	130702C_A3	130618G_A3	130521E_A3
Glass Ceramic	×	✓	○	-21.58	+0.02	-0.07	130709D_A1	130611G_A1	130528F_A1
Glass Mica	×	○	○	-4.61	+0.05	-0.44	130702C_A2	130618G_A2	130521E_A2
Machinable High-Alumina Ceramic	×	✓	○	-92.53	-0.89	+4.60	130709D_A2	130611G_A2	130528F_A2
Magnesium Oxide	×	✓	×	-4.05	N/A	-100.00	130425C_A1	130528G_A1	130604E_A1
Quartz	×	✓	✓	-100.00	-0.13	0.00	130412C_A1	130528G_A4	130321G_A1
Sapphire	×	✓	✓	-51.82	0.00	-0.01	130222C_A1	130311C_A1	130312E_A1
				-47.14	-0.03	0.00	130222C_A2	130311C_A2	130312E_A2
Sintered Alumina	×	✓	✓	-4.07	-0.05	-0.04	130222D_A1	130311C_A3	130312E_A3
Soda lime	×	○	○	-100.00	N/A	-1.43	130412C_A2	130528G_A3	130321G_A2
Yttria-stabilized Zirconia	○	✓	✓	-0.05	-0.04	-0.30	130425C_A2	130528G_A2	130604E_A2
Zirconia	✓	✓	○	-0.01	0.00	-0.10	130709D_A3	130611G_A3	130528F_A3

Table 3

Summary of bulk nitride ceramic samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Hot-Pressed Boron Nitride	×	○	○	-5.51	-0.54	-0.20	130321C_A2	130425D_A1	130416G_A4
Pyrolytic Boron Nitride	×	○	○	-19.77	-1.55	+0.84	130321C_A1	130425D_A2	130416G_A3
Silicon Nitride	×	✓	✓	-1.56	-0.02	-0.02	130321C_A3	130425D_A3	130416G_A2

Table 4

Summary of bulk carbide ceramic samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Silicon Carbide	○	✓	✓	-0.97	-0.05	0.00	130412C_A3	130425D_A6	130321G_A3
Tungsten Carbide	✓	✓	✓	0.00	0.00	-0.16	130723D_A1	130716E_A1	130709F_A2

Table 5

Summary of pure material samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers. Metals for which their alloy system was also analyzed are excluded from this table.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Aluminum	×		×	-100.00		-100.00	130618C_A2		090512G_A1
Germanium	×	○	×	N/A	-6.92	+42.33	130618C_A4	130702G_A3	130618E_A4
Gold	×	✓	✓	-8.76	+0.13	+0.09	130716C_A1	130723G_A1	090524F_A1
Iridium	×			-27.55			111027D_A1		
Lanthanum		×			+77.00			090630C_A1	
Magnesium	×		×	-27.28		-100.00	130618C_A5		090606E_A1
Niobium	○	○	○	+0.88	+0.71 -0.44	+0.89	090519B_A1	090630D_A1 130716G_A2	090429E_A1
Palladium	×	✓	✓	-55.17	+0.21	+0.97	130716C_A2	130723G_A2	090524E_A1
Scandium		✓			+3.18			090630B_A1	
Silicon	×	✓	×	N/A	-0.07	+1.53	130618C_A3	130702G_A2	130618E_A3
Silver	✓	✓	×	-0.04	0.00	-5.49	090519D_A1	130723G_A3	090429F_A1
Tantalum	○	○	×	+0.32	-5.40	+1.17	090519C_A1	130716G_A3	090512F_A1
Titanium	×	✓	○	+3.29 N/A	+0.20	+0.50	090611B_A1 130723C_A3	090530B_A1	130702E_A1
Vanadium	○	○	✓	+1.45	+1.29	-0.42	130723C_A1	130716G_A1	090429G_A1
Yttrium	×	×	×	-100.00	N/A	-100.00	090611C_A1	090530C_A1	130702E_A2 130723C_A2
Zirconium	×	✓	×	-0.28	+0.31	N/A	090611D_A1	090530D_A1	090512E_A1

Table 6

Summary of cobalt and cobalt alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Cobalt	✓	✓	×	+0.04	-0.01	-40.98	130604C_A1	130604G_A1	090606F_A1
						-41.54			130611F_A1
Co-W-Al Alloy	✓	✓	✓	-0.02	+0.07	-0.05	130604C_A2	130604G_A2	130611F_A2

Table 7

Summary of copper and copper alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Brass 260	×	○	×	-53.64	-9.29	-100.00	130521C_A3	130514G_A3	130425E_A3
Constantan (TC J-, TC E-)	○	✓	×	+2.00	-0.09	-100.00	130521C_A1	130514G_A1	130425E_A1
Copper	×	○	×	-100.00	-6.36	-100.00	130521C_A2	130514G_A2	130425E_A2

Table 8

Summary of iron and iron alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
1018 Steel	○	○	×	+2.58	+0.86	-66.95	130618C_A1	130702G_A1	130618E_A1
15-5 PH	○	○	×	+0.27	+0.88	-1.22	130514D_A1	130507E_A2	130416F_A2
17-4 PH	○	○	×	+0.25	+0.86	-1.96	130514D_A2	130507E_A3	130416F_A3
316L Stainless Steel	○	○	×	+0.81	+0.81	-0.08	130311D_A1	130507E_A1	130416F_A1
				+0.24	+2.46	+0.80	130311D_A2	130507E_A4	130416F_A4
				+0.27			130311D_A3		

Table 9

Summary of molybdenum and molybdenum alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Molybdenum	✓	✓	✓	+0.01	+0.03	0.00	130625D_A2	130625G_A2	090606G_A1
TZM	✓	✓	✓	0.00	0.00	0.00	130625D_A1	130625G_A1	131101E_A1

Table 11

Summary of platinum and platinum alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
Platinum (TC R-)	×	✓	✓	-100.00	+1.83	+1.63	130507C_A2	130507G_A3	130412E_A3
Platinum- Rhodium (TC R+)	×	✓	✓	-86.38	+1.70	+1.26	130507C_A3	130507G_A2	130412E_A2
Platinum- Ruthenium	✓	✓	✓	+2.27	+2.50	+5.06	130507C_A1	130507G_A1	130412E_A1

Table 12

Summary of tungsten and tungsten alloy samples including recommendation (good to excellent potential (✓), possible uses (○), not recommended (×)), relative weight change, and corresponding sample numbers.

Name	Recommendation			Weight Change (%)			Sample Nr.		
	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl	NH ₃ -Na	NH ₃	NH ₃ -Cl
High-Strength Durable Tungsten	✓	✓	○	-0.10	-0.01	-0.32	130611D_A2	130709G_A2	130625F_A2
Tungsten	✓	✓	○	-0.94	0.00	0.00 +0.05	130611D_A1	130709G_A1	090524G_A1 130625F_A1
Tungsten-Rhenium (TC C-)	✓	✓	✓	-0.02	-0.04	+0.10	130611D_A3	130709G_A3	130625F_A3
Tungsten-Rhenium (TC C+)	✓	✓	✓	-1.29	-0.04	+0.07	130611D_A4	130709G_A4	130625F_A4

Table A.1

Summary of performed runs wherein specified amounts of ammonia, sodium, and ammonium chloride were filled into an autoclave made of either Inconel 625 (Inc625) or René 41 (R41). Duration at specified external wall temperature with resulting peak pressure is provided.

Run Nr.	Reactor	Fill Amount (g)			Duration (days)	Peak	T _{external} ^a (°C)
	Material	NH ₃	Na	NH ₄ Cl		Pressure (MPa)	
090429E	Inc625	20.7		1.662	4	100	575
090429F	Inc625	20.7		1.618	4	96	575
090429G	Inc625	21.2		1.629	4	99	575
090512E	Inc625	20.7		1.622	4	102	575
090512F	Inc625	20.7		1.628	4	101	575
090512G	Inc625	20.8		1.620	4	99	575
090519B	R41	29.8	1.400		4	182	575
090519C	R41	29.9	1.400		4	178	575
090519D	R41	29.7	1.400		4	178	575
090524E	Inc625	20.7		1.620	4	95	575
090524F	Inc625	20.6		1.630	4	93	575
090524G	Inc625	21.6		1.620	4	103	575
090530B	R41	34.8			4	211	575
090530C	R41	34.3			4	203	575
090530D	R41	34.2			4	207	575
090606E	Inc625	20.6		1.640	4	86	575
090606F	Inc625	20.6		1.630	4	88	575
090606G	Inc625	20.4		1.640	4	93	575
090611B	R41	36.9	1.500		1*	236	575

090611C	R41	37.1	1.500		4	232	575
090611D	R41	36.9	1.500		4	237	575
090630B	R41	38.2			4	246	565
090630C	R41	38.4			3	256	565
090630D	R41	38.4			4	243	565
090713B	R41	36.5			6.25	243	575
090713C	R41	36.1			6.25	234	575
111027D	R41	22.80	1.500		5	174	575
130222C	R41	26.57	1.765		6	241	575
130222D	R41	26.56	1.770		6	218	575
130311C	R41	26.86			6	232	575
130311D	R41	26.77	1.800		6	250	575
130312E	Inc625	16.06		1.605	6	105	575
130321C	R41	20.49	1.800		6	249	525
130321G	Inc625	14.42		1.500	6	97	575
130412C	R41	21.26	1.414		6	208	545
130412D	R41	21.34	1.401		8*	48	575
130412E	Inc625	16.03		1.607	6	110	545
130416D	R41	21.36	1.401		6	171	575
130416F	Inc625	14.47		1.450	6	112	550
130416G	Inc625	14.52		1.450	6	100	575
130425C	R41	23.56	1.553		6	239	575
130425D	R41	26.57			6	224	575
130425E	Inc625	14.43		1.450	6	101	575
130507C	R41	23.53	1.550		6	182	575
130507E	Inc625	11.48			6	87	575
130507G	Inc625	15.27			6	101	575
130514D	R41	25.04	1.646		6	252	575

130514F	Inc625	14.50	1.449	6	99	575
130514G	Inc625	15.24		6	102	575
130521C	R41	25.04	1.663	6	199	575
130521E	Inc625	14.43	1.452	6	109	575
130521G	Inc625	15.25		6	103	575
130528D	R41	25.05	1.641	6	218	575
130528F	Inc625	13.67	1.371	6	99	575
130528G	Inc625	14.49		6	98	575
130604C	R41	25.04	1.657	6	207	575
130604E	Inc625	13.74	1.371	6	91	575
130604G	Inc625	14.50		6	104	575
130611D	R41	25.80	1.709	6	210	575
130611F	Inc625	13.74	1.376	6	100	575
130611G	Inc625	14.50		6	101	575
130618C	R41	25.80	1.704	6	234	575
130618E	Inc625	13.75	1.375	6	80	575
130618G	Inc625	14.49		6	103	575
130625D	R41	25.81	1.702	6	213	575
130625F	Inc625	13.74	1.372	6	95	575
130625G	Inc625	14.49		6	100	575
130702C	R41	25.81	1.702	6	260	540
130702E	Inc625	13.75	1.370	6	91	575
130702G	Inc625	14.51		3	110	575
130709D	R41	25.81	1.700	6	232	575
130709F	Inc625	13.75	1.366	6	89	575
130709G	Inc625	14.49		6	100	575
130716C	R41	25.81	1.704	6	236	575
130716E	Inc625	14.51		6	104	575

130716G	Inc625	14.51		6	98	575
130723C	R41	25.81	1.698	6	259	500
130723D	R41	25.81	1.701	6	218	575
130723G	Inc625	14.48		6	100	575
131101E	Inc625	11.47	1.137	12	85	575

*) Indicate runs during which a leak occurred.

a) Internal fluid temperature is estimated to be ~80 °C cooler

Table A.2

Summary of samples and their corresponding run (first 7 alphanumeric digits of the sample number) investigated in this study

Sample Nr.	Environment*	Name*	Nominal Chemical Composition	Type	Source	Product*	Purity (%)	Form	Sample Size (L x W x H) or (DIA x L) (all in mm)	Weight (g)	
										Before	After
090429E_A1	NH ₃ -Cl	Niobium	Nb	PC	Alfa Aesar	14358	99.8	Sheet	28 x 15 x 0.5	1.69636	1.71148
090429F_A1	NH ₃ -Cl	Silver	Ag	PC	Alfa Aesar	39181	99.9	Sheet	25 x 11 x 0.5	1.41843	1.34052
090429G_A1	NH ₃ -Cl	Vanadium	V	PC	Alfa Aesar	14633	99.5	Sheet	27 x 16 x 0.25	0.60827	0.6057
090512E_A1	NH ₃ -Cl	Zirconium	Zr	PC	Alfa Aesar	10591	99.8	Sheet	25 x 10 x 0.25	0.46185	0
090512F_A1	NH ₃ -Cl	Tantalum	Ta	PC	Alfa Aesar	10355	99.95 (ex. Nb)	Sheet	27 x 10 x 0.5	1.92269	1.94512
090512G_A1	NH ₃ -Cl	Aluminum	Al	PC	Alfa Aesar	40531	99.99	Sheet	25 x 10 x 0.5	0.42331	0
090519B_A1	NH ₃ -Na	Niobium	Nb	PC	Alfa Aesar	14358	99.8	Sheet	23 x 15 x 0.5	1.47592	1.48894
090519C_A1	NH ₃ -Na	Tantalum	Ta	PC	Alfa Aesar	10355	99.95 (ex. Nb)	Sheet	25 x 15 x 0.5	2.90061	2.90986
090519D_A1	NH ₃ -Na	Silver	Ag	PC	Alfa Aesar	39181	99.9	Sheet	25 x 14 x 0.5	1.75289	1.75224
090524E_A1	NH ₃ -Cl	Palladium	Pd	PC	Alfa Aesar	11514	99.9	Sheet	26 x 8 x 0.5	1.04162	1.05174
090524F_A1	NH ₃ -Cl	Gold	Au	PC	Alfa Aesar	36148	99.95	Sheet	27 x 9 x 0.5	2.07708	2.07902
090524G_A1	NH ₃ -Cl	Tungsten	W	PC	Alfa Aesar	10414	99.95	Sheet	25 x 12 x 0.5	2.64294	2.64291
090530B_A1	NH ₃	Titanium	Ti	PC	Alfa Aesar	43676	99.99+	Sheet	26 x 7 x 0.5	0.42967	0.43054
090530C_A1	NH ₃	Yttrium	Y	PC	Alfa Aesar	616	99.9	Sheet	24 x 6 x 0.64	0.42026	0
090530D_A1	NH ₃	Zirconium	Zr	PC	Alfa Aesar	10591	99.8	Sheet	24 x 17 x 0.25	0.6305	0.63243

090606E_A1	NH ₃ -Cl	Magnesium	Mg	PC	Alfa Aesar	40604	99.9	Sheet	24 x 8 x 1	0.37224	0
090606F_A1	NH ₃ -Cl	Cobalt	Co	PC	Alfa Aesar	42659	99.95	Sheet	27 x 9 x 0.5	1.061	0.62618
090606G_A1	NH ₃ -Cl	Molybdenum	Mo	PC	Alfa Aesar	10045	99.95	Sheet	27 x 10 x 0.5	1.39249	1.39251
090611B_A1	NH ₃ -Na	Titanium	Ti	PC	Alfa Aesar	43676	99.99+	Sheet	18 x 15 x 0.5	0.49011	0.50624
090611C_A1	NH ₃ -Na	Yttrium	Y	PC	Alfa Aesar	616	99.9	Sheet	17 x 11 x 0.64	0.56599	0
090611D_A1	NH ₃ -Na	Zirconium	Zr	PC	Alfa Aesar	10591	99.8	Sheet	17 x 12 x 0.25	0.60692	0.60524
090630B_A1	NH ₃	Scandium	Sc	PC	Alfa Aesar	39996	99.9	Pieces	between 1-5	2.103	2.1699
090630C_A1	NH ₃	Lanthanum	La	PC	Alfa Aesar	175	99.9	Pieces	between 1-8	4.166	7.37373
090630D_A1	NH ₃	Niobium	Nb	PC	Alfa Aesar	14358	99.8	Sheet	24 x 17 x 0.5	1.74535	1.75782
111027D_A1	NH ₃ -Na	Iridium	Ir	PC	Furuya Metal	1184269		Sheet	5 x 5 x 1	0.53691	0.38900
130222C_A1	NH ₃ -Na	Sapphire	Al ₂ O ₃	SC	Namiki	SSP		Sheet	10 x 10 x 0.4	0.18418	0.08873
130222C_A2		Sapphire	Al ₂ O ₃	SC	Namiki	DSP		Sheet	10 x 10 x 0.4	0.24157	0.12769
130222D_A1	NH ₃ -Na	Sintered Alumina	Al ₂ O ₃	PC	McDanel		99.8	Sheet	10 x 10 x 3	2.33888	2.2436
130311C_A1	NH ₃	Sapphire	Al ₂ O ₃	SC	Namiki	SSP		Sheet	12 x 6 x 0.4	0.12372	0.12372
130311C_A2		Sapphire	Al ₂ O ₃	SC	Namiki	DSP		Sheet	13 x 6 x 0.4	0.14239	0.14235
130311C_A3		Sintered Alumina	Al ₂ O ₃	PC	McDanel		99.8	Sheet	10 x 10 x 3	1.19212	1.19155
130311D_A1	NH ₃ -Na	316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	Swagelok			Tube	6.4 DIA x 100	12.14854	12.24657
130311D_A2		316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	OnlineMetals.com			Rod	6.4 DIA x 100	25.05155	25.11253
130311D_A3		316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	OnlineMetals.com			Rod	6.4 DIA x 100	24.89881	24.96595

130312E_A1		Sapphire	Al ₂ O ₃	SC	Namiki	SSP		Sheet	12 x 5 x 0.4	0.12322	0.12321
130312E_A2	NH ₃ -Cl	Sapphire	Al ₂ O ₃	SC	Namiki	DSP		Sheet	10 x 10 x 0.4	0.11996	0.11996
130312E_A3		Sintered Alumina	Al ₂ O ₃	PC	McDanel		99.8	Sheet	10 x 10 x 3	1.95348	1.95273
130321C_A1		Pyrolytic Boron Nitride	BN	PC	Momentive			Sheet	15 x 7 x 1	0.19078	0.15306
130321C_A2	NH ₃ -Na	Hot Pressed Boron Nitride	BN	PC	Saint Gobain			Sheet	13x 8 x 1.5	0.3511	0.33177
130321C_A3		Silicon Nitride	Si ₃ N ₄	PC	Bomas Machine Specialties			Rod	26 x 4 x 3	1.00132	0.98574
130321G_A1		Quartz	SiO ₂	SC	MTI Corp	DSP	99.99	Sheet	10 x 10 x 0.5	0.13116	0.13116
130321G_A2	NH ₃ -Cl	Soda lime	Si _{24.4} Al _{0.5} K _{0.6} Mg _{2.3} Na _{10.0} Ca _{2.5} O _{59.7}	A	Goldseal	3010		Sheet	10 x 8 x 1	0.20036	0.1975
130321G_A3		Silicon Carbide	SiC	SC	NovaSiC	SSP		Sheet	8 x 8 x 0.4	0.08593	0.08593
130412C_A1		Quartz	SiO ₂	SC	MTI Corp	DSP	99.99	Sheet	10 x 10 x 0.5	0.13441	0
130412C_A2	NH ₃ -Na	Soda lime	Si _{24.4} Al _{0.5} K _{0.6} Mg _{2.3} Na _{10.0} Ca _{2.5} O _{59.7}	A	Goldseal	3010		Sheet	10 x 7 x 1	0.1862	0
130412C_A3		Silicon Carbide	SiC	SC	NovaSiC	SSP		Sheet	9 x 7 x 0.4	0.0957	0.09477
130412E_A1		Platinum-Ruthenium	Pt ₉₁ Ru ₉	PC	Alfa Aesar	42686		Sheet	20 x 20 x 0.05	0.54379	0.57132
130412E_A2	NH ₃ -Cl	Platinum-Rhodium (TC R+)	Pt ₇₈ Rh ₂₂	PC	Omega	P13R-015		Wire	0.4 DIA x 150	0.5608	0.56787
130412E_A3		Platinum (TC R-)	Pt	PC	Omega	P13R-015		Wire	0.4 DIA x 64	0.6081	0.61804

Hafnium										
130416D_A1	NH ₃ -Na	Nitride coated	HfN / Al ₂ O ₃	C		Sheet	25 x 11 x 0.4	0.73843	0	
		Sapphire								
130416D_A2		Silicon Nitride	Si ₃ N ₄	PC	Bomas Machine Specialties	Rod	23 x 4 x 3	0.96198	0.88822	
130416F_A1	NH ₃ -Cl	316L Stainless	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4}	PC	OnlineMetals.co	Rod	6.3 DIA x 100	24.74875	24.72780	
		Steel	4C _{0.4} P _{0.1} S _{0.1}		m					
130416F_A2		15-5 PH	Fe _{75.9} Cr _{16.1} Ni _{4.5} Si _{2.0} Mn _{1.0} C _{0.3} N	PC	OnlineMetals.co	Rod	6.3 DIA x 75	18.30252	18.07854	
			b _{0.2} P _{0.1} S _{0.1}		m					
130416F_A3		17-4 PH	Fe _{75.6} Cr _{17.1} Ni _{3.7} Si _{1.9} Mn _{1.0} C _{0.3} N	PC	OnlineMetals.co	Rod	6.3 DIA x 72	17.68322	17.33642	
		b _{0.2} P _{0.1} S _{0.1}		m						
130416F_A4		316L Stainless	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4}	PC	Swagelok	Tube	6.3 DIA x 100	12.18927	12.28712	
		Steel	4C _{0.4} P _{0.1} S _{0.1}							
Hafnium										
130416G_A1	NH ₃ -Cl	Nitride coated	HfN / Al ₂ O ₃	C		Sheet	30 x 13 x 0.4	0.85264	0.85249	
		Sapphire								
130416G_A2		Silicon Nitride	Si ₃ N ₄	PC	Bomas Machine Specialties	Rod	23 x 4 x 3	0.9041	0.90389	
130416G_A3		Pyrolytic	BN	PC	Momentive	Sheet	13 x 7 x 1	0.15888	0.16021	
		Boron Nitride								
130416G_A4	Hot Pressed	BN	PC	Saint Gobain	Sheet	13x 10 x 1.5	0.42931	0.42843		
		Boron Nitride								
Magnesium										
130425C_A1	NH ₃ -Na		MgO	SC	MTI Corp	DSP	Sheet	10 x 10 x 0.5	0.18672	0.17916
		Oxide								
130425C_A2		Yttria-	Zr ₂₃ Y ₄ O ₅₂	SC	MTI Corp		Sheet	9 x 9 x 0.5	0.26417	0.26405

		stabilized Zirconia								
130425D_A1		Hot Pressed Boron Nitride	BN	PC	Saint Gobain		Sheet	13 x 10 x 1.5	0.44452	0.44212
130425D_A2		Pyrolytic Boron Nitride	BN	PC	Momentive		Sheet	15 x 7 x 1	0.17435	0.17165
130425D_A3		Silicon Nitride	Si ₃ N ₄	PC	Bomas Machine Specialties		Rod	23 x 4 x 3	0.94406	0.94388
130425D_A4	NH ₃	Hafnium Nitride coated Sapphire Titanium	HfN / Al ₂ O ₃	C			Sheet	30 x 7 x 0.4	0.27729	0.27726
130425D_A5		Nitride coated Sapphire	TiN / Al ₂ O ₃	C			Sheet	18 x 10 x 0.4	0.33621	0.33621
130425D_A6		Silicon Carbide	SiC	SC	NovaSiC	SSP	Sheet	9 x 9 x 0.4	0.10651	0.10646
130425E_A1		Constantan (TC J-, TC E-)	Cu ₅₃ Ni ₄₇	PC	Omega	IRCO-015	Wire	0.4 DIA x 64	0.80144	0
130425E_A2	NH ₃ -Cl	Copper	Cu	PC	Alfa Aesar	36395 99.998	Sheet	24 x 24 x 0.5	0.28907	0
130425E_A3		Brass 260	Cu ₇₁ Zn ₂₉	PC	Alfa Aesar	45181	Sheet	25 x 22 x 0.5	0.54517	0
130507C_A1		Platinum- Ruthenium	Pt ₉₁ Ru ₉	PC	Alfa Aesar	42686	Sheet	20 x 10 x 0.05	0.27841	0.28472
130507C_A2	NH ₃ -Na	Platinum (TC R-)	Pt	PC	Omega	P13R-015	Wire	0.4 DIA x 38	0.27741	0
130507C_A3		Platinum- Rhodium (TC	Pt ₇₈ Rh ₂₂	PC	Omega	P13R-015	Wire	0.4 DIA x 51	0.34255	0.04666

		R+)								
130507E_A1	NH ₃	316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	OnlineMetals.com		Rod	6.3 DIA x 100	24.79494	24.9951
130507E_A2		15-5 PH	Fe _{75.9} Cr _{16.1} Ni _{4.5} Si _{2.0} Mn _{1.0} C _{0.3} N _{b0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.com		Rod	6.3 DIA x 73	17.81681	17.97319
130507E_A3		17-4 PH	Fe _{75.6} Cr _{17.1} Ni _{3.7} Si _{1.9} Mn _{1.0} C _{0.3} N _{b0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.com		Rod	6.3 DIA x 73	17.91821	18.07289
130507E_A4		316L Stainless Steel	Fe _{64.7} Cr _{18.1} Ni _{11.3} Mn _{2.0} Si _{2.0} Mo _{1.4} C _{0.4} P _{0.1} S _{0.1}	PC	Swagelok		Tube	6.3 DIA x 100	11.9654	12.26012
130507G_A1	NH ₃	Platinum-Ruthenium	Pt ₉₁ Ru ₉	PC	Alfa Aesar	42686	Sheet	20 x 10 x 0.05	0.25308	0.25941
130507G_A2		Rhodium (TC R+)	Pt ₇₈ Rh ₂₂	PC	Omega	P13R-015	Wire	0.4 DIA x 51	0.55248	0.56187
130507G_A3		Platinum (TC R-)	Pt	PC	Omega	P13R-015	Wire	0.4 DIA x 76	0.60912	0.62025
130514D_A1	NH ₃ -Na	15-5 PH	Fe _{75.9} Cr _{16.1} Ni _{4.5} Si _{2.0} Mn _{1.0} C _{0.3} N _{b0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.com		Rod	6.3 DIA x 74	18.13513	18.18329
130514D_A2		17-4 PH	Fe _{75.6} Cr _{17.1} Ni _{3.7} Si _{1.9} Mn _{1.0} C _{0.3} N _{b0.2} P _{0.1} S _{0.1}	PC	OnlineMetals.com		Rod	6.3 DIA x 74	18.20757	18.25328
130514F_A1	NH ₃ -Cl	Chromel C (TC K+) / Alumel (TC K-) Junction	Ni ₈₉ Cr ₁₁ / Ni ₉₂ Mn ₂ Al ₄ Si ₂	J	Omega	KMQIN	Wire	0.5 DIA x 101	3.24486	1.18063
130514F_A2		Nicrosil (TC	Ni _{81.2} Cr _{15.7} Si _{2.8} Mg _{0.2} /	J	Omega	NMQXL	Wire	0.5 DIA x 101	3.52521	1.43892

		N+) / Nisil (TC N-) Junction	Ni _{91.2} Si _{8.8}								
130514G_A1	NH ₃	Constantan (TC J-, TC E-)	Cu ₅₃ Ni ₄₇	PC	Omega	IRCO-015		Wire	0.4 DIA x 64	0.76539	0.76472
130514G_A2		Copper	Cu	PC	Alfa Aesar	36395	99.998	Sheet	24 x 24 x 0.5	0.28804	0.26972
130514G_A3		Brass 260	Cu ₇₁ Zn ₂₉	PC	Alfa Aesar	45181		Sheet	25 x 22 x 0.5	0.56945	0.51656
130521C_A1	NH ₃ -Na	Constantan (TC J-, TC E-)	Cu ₅₃ Ni ₄₇	PC	Omega	IRCO-015		Wire	0.4 DIA x 64	0.79359	0.8095
130521C_A2		Copper	Cu	PC	Alfa Aesar	36395	99.998	Sheet	24 x 24 x 0.5	0.20301	0
130521C_A3		Brass 260	Cu ₇₁ Zn ₂₉	PC	Alfa Aesar	45181		Sheet	25 x 22 x 0.5	0.51921	0.24072
130521E_A1	NH ₃ -Cl	Alumina Silicate	Al _{13.1} Si _{21.0} Fe _{0.8} K _{0.6} Ti _{0.4} O _{64.1}	PC	McMaster	8479K41		Sheet	31 x 16 x 6	7.71861	7.72682
130521E_A2		Glass Mica	Si _{15.4} Mg _{8.5} F _{4.2} Al _{3.2} K _{2.1} B _{2.0} O _{64.5}	A	McMaster	8489K61		Sheet	30 x 15 x 7	7.33903	7.3069
130521E_A3		Fused Silica	SiO ₂	A	McMaster	1357T41	99.995	Sheet	12 DIA x 1.6	0.43824	0.43429
130521G_A1		Nickel	Ni	PC	Alfa Aesar	44824	99.5	Sheet	100 x 12 x 1	10.60294	10.60829
130521G_A2	NH ₃	Chromel C (TC K+) / Alumel (TC K-) Junction	Ni ₈₉ Cr ₁₁ / Ni ₉₂ Mn ₂ Al ₄ Si ₂	J	Omega	KMQIN		Wire	0.5 DIA x 101	3.24927	3.26095
130521G_A3		Nicrosil (TC N+) / Nisil (TC N-) Junction	Ni _{81.2} Cr _{15.7} Si _{2.8} Mg _{0.2} / Ni _{91.2} Si _{8.8}	J	Omega	NMQXL		Wire	0.5 DIA x 101	3.61343	3.62358
130528D_A1		Nickel	Ni	PC	Alfa Aesar	44824	99.5	Sheet	100 x 12 x 1	10.62452	10.62556
130528D_A2	NH ₃ -Na	Chromel C (TC K+) / Alumel	Ni ₈₉ Cr ₁₁ / Ni ₉₂ Mn ₂ Al ₄ Si ₂	J	Omega	KMQIN		Wire	0.5 DIA x 101	3.24131	3.24205

		(TC K-) Junction Nicrosil (TC N+) / Nisil (TC N-) Junction	$\text{Ni}_{81.2}\text{Cr}_{15.7}\text{Si}_{2.8}\text{Mg}_{0.2} /$ $\text{Ni}_{91.2}\text{Si}_{8.8}$	J	Omega	NMQXL		Wire	0.5 DIA x 101	3.53565	3.53678
130528F_A1		Glass Ceramic	$\text{Si}_{20.7}\text{Al}_{7.9}\text{Li}_{3.2}\text{Mg}_{1.9}\text{Ca}_{1.4}\text{Na}_{0.6}$ $\text{Ti}_{0.6}\text{Zn}_{0.6}\text{Ba}_{0.5}\text{K}_{0.4}\text{Zr}_{0.4}\text{O}_{61.8}$	A	McMaster	84815K49		Sheet	40 x 10 x 5	6.35095	6.34659
130528F_A2	NH ₃ -Cl	Machinable High Alumina Ceramic	Al_2O_3	PC	McMaster	8978T61	99	Sheet	38 x 8 x 1.5	1.32229	1.38315
130528F_A3		Zirconia	ZrO_2	PC	McMaster	8750K37	97	Rod	6.3 DIA x 50	9.2357	9.22606
130528G_A1		Magnesium Oxide	MgO	SC	MTI Corp	DSP		Sheet	10 x 10 x 0.5	0.1866	--
130528G_A2	NH ₃	Yttria- stabilized Zirconia	$\text{Zr}_{23}\text{Y}_4\text{O}_{52}$	SC	MTI Corp			Sheet	10 x 10 x 0.5	0.27987	0.27977
130528G_A3		Soda lime	$\text{Si}_{24.4}\text{Al}_{0.5}\text{K}_{0.6}\text{Mg}_{2.3}\text{Na}_{10.0}\text{Ca}_{2.5}$ $\text{O}_{59.7}$	A	Goldseal	3010		Sheet	8 x 8 x 1	0.17734	--
130528G_A4		Quartz	SiO_2	SC	MTI Corp	DSP	99.99	Sheet	10 x 8 x 0.5	0.10542	0.10528
130604C_A1		Cobalt	Co	PC	Alfa Aesar	42659	99.95	Sheet	14 x 10 x 0.5	0.66617	0.66642
130604C_A2	NH ₃ -Na	Co-W-Al Alloy	$\text{Co}_{80}\text{W}_{10.6}\text{Al}_{9.4}$	PC	UCSB			Ingot	14 x 14 x 1	1.90086	1.90048
130604C_A3		Hastelloy C- 276	$\text{Ni}_{58.9}\text{Cr}_{18.7}\text{Mo}_{10.4}\text{Fe}_{6.2}\text{Co}_{2.7}\text{W}_{1.3}$ $\text{Mn}_{1.1}\text{V}_{0.4}\text{Si}_{0.2}\text{P}_{0.1}\text{S}_{0.1}\text{C}_{0.1}$	PC	Fry Steel	Fry Alloy C 276		Rod	6.3 DIA x 74	20.96741	20.97154
130604E_A1	NH ₃ -Cl	Magnesium Oxide	MgO	SC	MTI Corp	DSP		Sheet	10 x 10 x 0.5	0.17599	0

130604E_A2		Yttria-stabilized Zirconia	$Zr_{23}Y_4O_{52}$	SC	MTI Corp			Sheet	10 x 9 x 0.5	0.28586	0.28501
130604G_A1		Cobalt	Co	PC	Alfa Aesar	42659	99.95	Sheet	18 x 7 x 0.5	0.55082	0.55079
130604G_A2	NH ₃	Co-W-Al Alloy	$Co_{80}W_{10.6}Al_{9.4}$	PC	UCSB			Ingot	9 x 9 x 0.8	0.7205	0.72104
130604G_A3		Hastelloy C-276	$Ni_{58.9}Cr_{18.7}Mo_{10.4}Fe_{6.2}Co_{2.7}W_{1.3}Mn_{1.1}V_{0.4}Si_{0.2}P_{0.1}S_{0.1}C_{0.1}$	PC	Fry Steel	Fry Alloy C 276		Rod	6.3 DIA x 74	20.55137	20.56195
130611D_A1		Tungsten	W	PC	Alfa Aesar	10414	99.95	Sheet	48 x 17 x 0.5	4.31817	4.27769
130611D_A2		High-Strength Durable Tungsten	$W_{74.7}Ni_{15.6}Cu_{9.6}$	PC	McMaster	8279k15		Rod	6.3 DIA x 49	29.68264	29.6541
130611D_A3	NH ₃ -Na	Tungsten-Rhenium (TC C-)	$W_{74}Re_{26}$	PC	Omega	T5R-010		Wire	0.25 DIA x 80	0.32771	0.32764
130611D_A4		Tungsten-Rhenium (TC C+)	$W_{95}Re_5$	PC	Omega	T5R-010		Wire	0.25 DIA x 110	0.32562	0.32143
130611F_A1		Cobalt	Co	PC	Alfa Aesar	42659	99.95	Sheet	18 x 10 x 0.5	0.71897	0.42031
130611F_A2		Co-W-Al Alloy	$Co_{80}W_{10.6}Al_{9.4}$	PC	UCSB			Ingot	14 x 14 x 1.6	3.32139	3.3196
130611F_A3	NH ₃ -Cl	Nickel	Ni	PC	Alfa Aesar	44824	99.5	Sheet	101 x 12 x 1	10.65129	5.08878
130611F_A4		Hastelloy C-276	$Ni_{58.9}Cr_{18.7}Mo_{10.4}Fe_{6.2}Co_{2.7}W_{1.3}Mn_{1.1}V_{0.4}Si_{0.2}P_{0.1}S_{0.1}C_{0.1}$	PC	Fry Steel	Fry Alloy C 276		Rod	6.3 DIA x 85	23.60138	23.59985
130611G_A1	NH ₃	Glass Ceramic	$Si_{20.7}Al_{7.9}Li_{3.2}Mg_{1.9}Ca_{1.4}Na_{0.6}Ti_{0.6}Zn_{0.6}Ba_{0.5}K_{0.4}Zr_{0.4}O_{61.8}$	A	McMaster	84815K49		Sheet	50 x 10 x 5	6.36584	6.36729
130611G_A2		Machinable	Al ₂ O ₃	PC	McMaster	8978T61	99	Sheet	48 x 9 x 1.5	1.3535	1.34144

		High Alumina									
		Ceramic									
130611G_A3		Zirconia	ZrO ₂	PC	McMaster	8750K37	97	Rod	6.3 DIA x 48	9.11963	9.1197
130618C_A1	NH ₃ -Na	1018 Steel	Fe _{98.3} Mn _{0.8} C _{0.9}	PC	Unknown			Sheet	19 x 13 x 0.9	3.39116	3.47881
130618C_A2		Aluminum	Al	PC	Alfa Aesar	40531	99.99	Sheet	23 x 12 x 0.5	0.13663	0
130618C_A3		Silicon	Si	SC	MTI Corp			Sheet	47 x 16 x 0.5	0.86671	--
130618C_A4		Germanium	Ge	SC	MTI Corp			Sheet	48 x 15 x 0.5	2.10573	--
130618C_A5		Magnesium	Mg	PC	Alfa Aesar	40604	99.9	Sheet	16 x 14 x 1	0.41244	0.29993
130618E_A1	NH ₃ -Cl	1018 Steel	Fe _{98.3} Mn _{0.8} C _{0.9}	PC	Unknown			Sheet	20 x 13 x 0.9	3.38799	1.11986
130618E_A2		Aluminum	Al	PC	Alfa Aesar	40531	99.99	Sheet	23 x 12 x 0.5	0.12983	0
130618E_A3		Silicon	Si	SC	MTI Corp			Sheet	30 x 18 x 0.5	0.69793	0.70861
130618E_A4		Germanium	Ge	SC	MTI Corp			Sheet	30 x 15 x 0.5	1.59984	2.27709
130618G_A1	NH ₃	Aluminum									
		Silicate	Al _{13.1} Si _{21.0} Fe _{0.8} K _{0.6} Ti _{0.4} O _{64.1}	PC	McMaster	8479K41		Sheet	30 x 15 x 6	7.67459	7.63696
		Glass Mica	Si _{15.4} Mg _{8.5} F _{4.2} Al _{3.2} K _{2.1} B _{2.0} O _{64.5}	A	McMaster	8489K61		Sheet	30 x 15 x 6	7.11173	7.11556
130618G_A3		Fused Silica	SiO ₂	A	McMaster	1357T41	99.995	Sheet	12 DIA x 1.6	0.43108	0.43099
130625D_A1	NH ₃ -Na	Titanium-									
		Zirconium-									
		Molybdenum	Mo _{99.6} Ti _{0.3} Zr _{0.1}	PC	Plansee			Rod	6.3 DIA x 67	16.07167	16.07175
		(TZM)									
130625D_A2		Molybdenum	Mo	PC	Alfa Aesar	10045	99.95	Sheet	23 x 22 x 0.5	1.5448	1.54491
130625F_A1	NH ₃ -Cl	Tungsten	W	PC	Alfa Aesar	10414	99.95	Sheet	33 x 16 x 0.5	2.87509	2.87643
130625F_A2		High-Strength Durable	W _{74.7} Ni _{15.6} Cu _{9.6}	PC	McMaster	8279k15		Rod	6.3 DIA x 49	29.59689	29.502

		Tungsten									
		Tungsten-									
130625F_A3		Rhenium (TC C-)	W ₇₄ Re ₂₆	PC	Omega	T5R-010		Wire	0.25 DIA x 80	0.32017	0.3205
		Tungsten-									
130625F_A4		Rhenium (TC C+)	W ₉₅ Re ₅	PC	Omega	T5R-010		Wire	0.25 DIA x 110	0.31876	0.31898
		Titanium-									
130625G_A1	NH ₃	Zirconium-Molybdenum (TZM)	Mo _{99.6} Ti _{0.3} Zr _{0.1}	PC	Plansee			Rod	6.3 DIA x 68	16.12363	16.12395
130625G_A2		Molybdenum	Mo	PC	Alfa Aesar	10045	99.95	Sheet	23 x 23 x 0.5	1.59113	1.59165
130702C_A1		Aluminum Silicate	Al _{13.1} Si _{21.0} Fe _{0.8} K _{0.6} Ti _{0.4} O _{64.1}	PC	McMaster	8479K41		Sheet	30 x 13 x 8	7.5337	7.41185
130702C_A2	NH ₃ -Na	Glass Mica	Si _{15.4} Mg _{8.5} F _{4.2} Al _{3.2} K _{2.1} B _{2.0} O _{64.5}	A	McMaster	8489K61		Sheet	30 x 15 x 6	7.33845	7.00029
130702C_A3		Fused Silica	SiO ₂	A	McMaster	1357T41	99.995	Sheet	12 DIA x 1.6	0.43215	0
130702E_A1		Titanium	Ti	PC	Alfa Aesar	43676	99.99+	Sheet	15 x 12 x 0.5	0.38914	0.39107
130702E_A2	NH ₃ -Cl	Yttrium	Y	PC	Alfa Aesar	616	99.9	Sheet	15 x 14 x 0.6	0.60065	0
130702G_A1		1018 Steel	Fe _{98.3} Mn _{0.8} C _{0.9}	PC	Unknown			Sheet	39 x 13 x 0.9	3.50269	3.53286
130702G_A2	NH ₃	Silicon	Si	SC	MTI Corp			Sheet	30 x 18 x 0.5	0.65774	0.65729
130702G_A3		Germanium	Ge	SC	MTI Corp			Sheet	35 x 13 x 0.5	1.72135	1.60227
130709D_A1		Glass Ceramic	Si _{20.7} Al _{7.9} Li _{3.2} Mg _{1.9} Ca _{1.4} Na _{0.6}	A	McMaster	84815K49		Sheet	50 x 10 x 5	6.57817	5.15855
	NH ₃ -Na		Ti _{0.6} Zn _{0.6} Ba _{0.5} K _{0.4} Zr _{0.4} O _{61.8}								
130709D_A2		Machinable High Alumina	Al ₂ O ₃	PC	McMaster	8978T61	99	Sheet	50 x 12 x 1.5	1.67698	0.12533

		Ceramic									
130709D_A3		Zirconia	ZrO ₂	PC	McMaster	8750K37	97	Rod	6.3 DIA x 48	9.25769	9.25703
		Zirconium									
130709F_A1		Nitride coated	ZrN / Zr	C				Sheet	50 x 15 x 0.3	1.45324	0.05896
	NH ₃ -Cl	Zirconium									
130709F_A2		Tungsten Carbide	(WC) ₉₀₋₉₄ Co ₆₋₁₀ Fe _{0.4}	PC	McMaster	8788A163		Rod	6.3 DIA x 50	22.69704	22.66184
130709G_A1		Tungsten	W	PC	Alfa Aesar	10414	99.95	Sheet	32 x 17 x 0.5	3.26742	3.26734
		High-Strength									
130709G_A2		Durable	W _{74.7} Ni _{15.6} Cu _{9.6}	PC	McMaster	8279k15		Rod	6.3 DIA x 48	28.79129	28.7892
		Tungsten									
		Tungsten-									
130709G_A3	NH ₃	Rhenium (TC C-)	W ₇₄ Re ₂₆	PC	Omega	T5R-010		Wire	0.25 DIA x 80	0.32317	0.32304
		Tungsten-									
130709G_A4		Rhenium (TC C+)	W ₉₅ Re ₅	PC	Omega	T5R-010		Wire	0.25 DIA x 110	0.31836	0.31824
130716C_A1		Gold	Au	PC	Alfa Aesar	36148	99.95	Sheet	15 x 8 x 0.5	1.17406	1.07121
130716C_A2	NH ₃ -Na	Palladium	Pd	PC	Alfa Aesar	11514	99.9	Sheet	15 x 12 x 0.5	1.2353	0.55373
130716E_A1		Tungsten Carbide	(WC) ₉₀₋₉₄ Co ₆₋₁₀ Fe _{0.4}	PC	McMaster	8788A163		Rod	6.3 DIA x 45	22.98623	22.98646
	NH ₃	Zirconium									
130716E_A2		Nitride coated	ZrN / Zr	C				Sheet	50 x 19 x 0.3	1.56085	1.54407
		Zirconium									
130716G_A1	NH ₃	Vanadium	V	PC	Alfa Aesar	14633	99.5	Sheet	36 x 18 x 0.14	0.54602	0.55307

130716G_A2		Niobium	Nb	PC	Alfa Aesar	14358	99.8	Sheet	46 x 17 x 0.6	3.85528	3.83817
130716G_A3		Tantalum	Ta	PC	Alfa Aesar	10355	99.95 (ex. Nb)	Sheet	30 x 23 x 0.5	6.81818	6.44971
130723C_A1		Vanadium	V	PC	Alfa Aesar	14633	99.5	Sheet	36 x 18 x 0.14	0.55322	0.56126
130723C_A2		Yttrium	Y	PC	Alfa Aesar	616	99.9	Sheet	24 x 13 x 0.7	0.88641	0
130723C_A3	NH ₃ -Na	Titanium	Ti	PC	Alfa Aesar	43676	99.99+	Sheet	20 x 14 x 0.3	0.3666	--
		Zirconium									
130723C_A4		Nitride coated	ZrN / Zr	C				Sheet	19 x 14 x 0.3	0.50199	0.03198
		Zirconium									
130723D_A1		Tungsten		PC	McMaster	8788A163		Rod	6.3 DIA x 48	22.99298	22.99294
		Carbide	(WC) ₉₀₋₉₄ Co ₆₋₁₀ Fe ₀₋₄								
	NH ₃ -Na	Zirconium									
130723D_A2		Nitride coated	ZrN / Zr	C				Sheet	30 x 17 x 0.3	1.56317	1.59069
		Zirconium									
130723G_A1		Gold	Au	PC	Alfa Aesar	36148	99.95	Sheet	14 x 7 x 0.5	0.8215	0.82257
130723G_A2	NH ₃	Palladium	Pd	PC	Alfa Aesar	11514	99.9	Sheet	16 x 11 x 0.5	1.23286	1.23547
130723G_A3		Silver	Ag	PC	EPSI Metals	Knd3023	99.99	Rod	18 x 10 x 9	16.73662	16.73661
		Titanium-									
131101E_A1	NH ₃ -Cl	Zirconium-		PC	Plansee			Rod	6.3 DIA x 38	6.83492	6.83523
		Molybdenum	Mo _{99.6} Ti _{0.3} Zr _{0.1}								
		(TZM)									

* Abbreviations: Polycrystalline (PC), Single Crystal (SC), Coating (C), Junction (J), Amorphous (A), Single Side Polished (SSP), Double Side Polished (DSP), Thermocouple wire (TC) from indicated type and lead (+/-)

* Environments: Autoclave filled with samples(s) and ammonia (NH_3), ammonia and sodium metal ($\text{NH}_3\text{-Na}$), or ammonia and ammonium chloride ($\text{NH}_3\text{-Cl}$)