Room Temperature Stress-Strain Hysteresis in Ti₂AlC Revisited

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ABSTRACT

Room temperature cyclic compressive testing of high purity fine (FG) and coarse (CG) grained Ti₂AlC were carried out in combination with Resonant Ultrasound Spectroscopy (RUS) tests and post mortem Electron-backscatter Diffraction (EBSD). The results show that the room temperature mechanical response of Ti₂AlC can be divided in four stress regions with distinct underlying mechanisms: (a) Region I (FG: 0 to ≈ 175 MPa; CG: 0 to ≈ 100 MPa) characterized by a linear elastic behavior; (b) Region II (FG: from ≈ 175 to ≈ 350 MPa; CG from ≈ 100 to ≈ 200 MPa) in which stress strain hysteric behavior due to reversible dislocation flow in the soft grains; (c) Region III (FG: from ≈ 350 to ≈ 1100 MPa; CG from ≈ 200 to ≈ 650 MPa) accumulation of dislocation walls (DWs) results in cyclic hardening and contribute to larger hysteretic loops and thus a larger energy dissipation per loading cycle than in Region II; (d) Region IV (FG: from ≈ 1100 MPa to failure, CG from ≈ 600 MPa to failure) characterized by the occurrence of microcracking that, in addition to the other aforementioned deformation mechanisms, contributes to the energy dissipated in each loading cycle.

KEYWORDS: Carbides, Damping capacity, Anisotropy, Yield behavior, Dislocation boundaries.

1. INTRODUCTION

The MAX phases are ternary carbides and nitrides with a nano-laminated hexagonal structure (space group $P63/mmc$) whose unique combination of metallic and ceramic-like properties have sparked great interest since the first MAX phase, namely Ti_3SiC_2 , was synthesized in bulk [1-3]. In general, these phases are potentially useful materials for many applications because some of them have high stiffness, strength, thermal shock resistance, good electrical and thermal conductivity combined with low density, hardness, and ease of machinability [3, 4]. Out of more than 70+ different MAX phases known today, Ti_2AlC is considered one of the best candidates for high temperature applications mostly because of its excellent oxidation resistance due to the formation of spallation-resistant and self-healing protective Al_2O_3 layer on surfaces exposed to air and/or water vapor [1, 4-8].

The unique mechanical properties of Ti_2AlC , and to the further extent of all other MAX phases, can be attributed to their plastically anisotropic, nano-laminated structure, in which numerous dislocations form and easily glide even at room temperature, but only on the basal planes. Furthermore, basal plane dislocations arrange themselves either in pileups along slip planes or as dislocation walls normal to the basal planes. Consequently, the MAX phases have fewer than five independent slip systems and therefore do not behave at room temperature as ductile materials in the classical sense [4]. However, what makes their mechanical behavior different from other brittle solids with limited number of active slip systems is their ability to deform additionally by buckling, kinking and kink band, (KB) formation [9-12]. First described by Orowan [9] and later by Frank and Stroh [10], kinks form in areas of maximum shear stress and consist of regularly spaced basal plane dislocations arranged in two dislocation walls (DWs) of opposite signs [10].

Both kinking and dislocation glide only in basal planes lead to the highly anisotropic plastic behavior of MAX phases where the orientation of the individual grain relative to the applied load dictates its mechanical response [4, 13, 14]. Therefore, it has been postulated that when basal planes are oriented at approximately 45° to the loading direction in compression - with high Schmid factors hereafter referred to as soft grains (green grains in **Figure 1**) – they

can easily deform by glide of basal plane dislocations once the local stress in the grain reaches the critical resolved shear stress (CRSS). However, grains whose basal planes are parallel to the loading direction, hereinafter referred to as longitudinal hard grains (blue grains in **Figure 1**), can deform by kinking once the local stress reaches a critical value [10], while the grains whose basal planes are perpendicular to the loading direction, hereinafter referred to as transversal hard grains (red grains in **Figure 1**), can hardly deform plastically and will only see elastic deformation.

One of the most intriguing properties of the MAX phases is their hysteretic stress-strain behavior that was reported for the first time in Ti_3SiC_2 during cyclic tension and compression loading, and later in other MAX phases such as Ti_2AlC , Ti_3AlC_2 , and Ti_3GeC_2 among others [15-19]. When these phases are subjected to cyclic loading, at room temperature, their stress-strain response is non-linear elastic (see **Figure 2**), and in some MAX phases such as Ti_2AlC , all but the first stress-strain hysteresis loops are closed, reversible and reproducible [17]. The amount of mechanical energy dissipated during each loading-unloading cycle is quite impressive for stiff solids like MAX phases, i.e. about 25% and 24% for Ti_3SiC_2 and Ti_2AlC ¹ per loading cycle [16, 20].

The first attempt to explain this hysteretic behavior, was a microscale dislocation model for kinking nonlinear elastic (KNE) deformation proposed by Barsoum et al. usually referred to as the KNE model [21-24]. The KNE model builds on the theory of Frank and Stroh's, who laid the theoretical foundation for kinking. According to the KNE model damping in the MAX phases occurs due to reversible formation and growth of incipient kink bands (IKBs), and their annihilation during unloading. IKBs consist of multiple coaxial dislocation loops stacked in parallel basal planes on the top of each other (see Figure 4a in [4]). IKBs are proposed to be precursors to fully developed kink bands (KBs) as their growth and dissociation result in the formation of two dislocation walls of opposite signs, usually referred to as mobile dislocation walls (MDWs). According to the KNE model, IKBs should form in the longitudinal hard grains (or kinking grains) under loading once the applied stress reaches a threshold value. When the

¹ Those values were estimated from [9, 25] for Ti_3SiC_2 and Ti_2AlC , respectively.

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4 stress is removed, dislocation loops forming the IKBs shrink back and annihilate, resulting in
5 permanent recovery of deformation strain and energy dissipation [25], **Figure 1b**.

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8 The KNE model predicts that the dissipated energy per unit volume per cycle (W_d) and
9 the non-linear elastic strain (ϵ_{NL}) should be linear function of σ^2 , where σ is the maximum applied
10 stress per cycle, while W_d should be a linear function of ϵ_{NL} . These relationships are in good
11 agreement with experimental observations. Moreover, the underlying micromechanism of the
12 KNE model offers an effective explanation for other experimental observations such that W_d
13 increases with grain size and porosity [20, 26, 27]. The KNE model, however does not account
14 for the contribution of eventual reversible deformation in any but kinking grains, which raises
15 an issue with the strain compatibility in polycrystalline structures. In other words, deformation
16 of the grains in which IKBs form due to their favorable orientation relative to applied stress has
17 to be accommodated by elastic or plastic deformation of surrounding grains not favorably
18 oriented for kinking, or otherwise large incompatibility stresses in neighboring grains and
19 eventual intergranular cracking (Zener-Stroh type cracks [28-30]) would appear, resulting in
20 hysteresis loops that are not fully closed and reproducible after a large number of loading
21 cycles. In addition, this model does not address the fact that the first loading-unloading
22 hysteresis loop might be open. Furthermore, one of the challenges of the KNE model is that
23 although permanent KB have been observed in the MAX phases, there are no documented
24 observations of IKBs [31].

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27 Recently, an alternate model with a radically different underlying mechanism was
28 proposed to explain the nonlinear hysteretic behavior of MAX phases, further referred to as
29 microcracking model (MC) [32]. This model is based on the work of Walsh [33] and of Lawn and
30 Marshall [34] and proposes that damage (i.e. microcracks and delamination) accumulation and
31 resulting friction between the delamination crack faces (**Figure 1c**) cause the observed
32 hysteretic behavior in MAX phases. Nevertheless, contrary to many previous observations Poon
33 et al. reported that Ti_2AlC initially behaved as linear elastic solid showing hysteretic loops only
34 beyond a “yield point” of 540 MPa [32]. The crack density calculated using this model was
35 found to be in agreement with crack densities measured using scanning electron microscopy
36 (SEM) images taken after loading to various strain levels. This model captures the nonlinearity

of the stress-strain behavior for loading above 540 MPa, and can potentially explain opening of the first loop due to microcracking. However, upon careful examination of the micrographs presented in that study, significant amounts of intergranular secondary phase (most likely TiAl_x) can be observed and neither their existence nor their potential effect on the mechanical response are addressed in that study. In addition, it is very unlikely that microcracking would result in stable and reproducible hysteretic loops for up to 100 cycles that have been reported for the MAX phases elsewhere [16].

A most recent study by Jones et al. [35], hereafter referred to as the reversible flow (RF) model, also disregards IKBs formation and annihilation as the main hysteretic mechanism in the MAX phases based on the interpretation of results obtained by: (i) in-situ synchrotron X-ray diffraction during cyclic compression testing; and (ii) elasto-plastic self-consistent (EPSC) modeling [36-38] that is derived from Eshelby's homogeneous inclusion method [39, 40]. According to this model, the macroscopically observed hysteretic behavior is attributed to plastic anisotropy or a Bauschinger-like effect. More specifically, this model proposes that the residual elastic lattice strains that buildup in hard grains upon the initial loading-unloading cycle as a result of anisotropic deformation and easy yielding and flow of only soft grains, are sufficient to drive reverse flow of soft grains, giving rise to some permanent plastic strain in the first loading-unloading cycle and reversible hysteretic behavior in subsequent loading cycles (**Figure 1d**). Although not explicitly stated in the original work by Jones et al. [35], the flow in soft grains is assumed to occur predominantly by reversible dislocation glide in basal planes to and from dislocation pile-ups onto the grain boundaries. Furthermore, the authors of the RF model argue against the concept of IKB formation due to the fact that their formation would not lead by itself to residual (plastic) strains and observed opening of the hysteresis loop during the first load-unload cycle. Although their numerical predictions using the EPSC model capture some of the qualitative details of the experimental responses, they do not quantitatively predict or explain all of the features observed in their mechanical tests. For example, predicted residual (plastic) strains after the first loading-unloading cycle up to 400 MPa are significantly larger than the measured ones (compare Figures 2 and 5 in [35]). In addition, contrary to many experimental observations [17, 20, 21, 31, 41, 42], the RF model predicts that W_d should be a

linear function of σ . Last but not least, although the RF model takes into account incompatibility stresses that develop due to plastic anisotropy, this model also attributes energy dissipation during cyclic loading exclusively to one mechanism, namely reversible dislocation flow in soft grains. The latter is in disagreement with some previous studies showing that the energy dissipated only by reversible glide of dislocations is insufficient to account for W_d obtained in experimental work [43].

Building on the RF model, Guitton et al. [14] used in-situ neutron diffraction to study the response of another MAX phase, namely Ti_2AlN , showing hysteretic behavior under cyclic compressive loading. Their results demonstrate the development of heterogeneous strains as a result of anisotropic elastic and plastic deformation during loading that is even more pronounced above a threshold stress of around 200 MPa, when the mechanical response of Ti_2AlN becomes hysteretic. Moreover, deformation in grains with basal planes parallel to the applied load (longitudinal hard grains) was found to be most likely elastic at all applied stresses, as proposed by the RF model. However, contrary to the RF model, results of this work indicate that when the applied stress exceeds a critical value of around 200 MPa, reversible “plastic” deformation occurs not only in soft grains, but also in transversal hard grains; more specifically in all grains in which the angle between the [0001] direction and the direction of applied stress is below approximately 70° . The latter is attributed to the plastic bending of transversal hard grains caused by complex multi-axial stress states and complex interconnections of grains in a polycrystalline structure. In the most recent *in situ* neutron diffraction study of highly textured Ti_2AlC during cyclic compression (i.e. with basal planes mostly normal or perpendicular to the loading direction), Sharma et al. [44] also showed development of heterogeneous lattice strains once the yield point is reached in the soft grains. However, their calculations of dislocation densities based on the full width of the half maxima (FWHM) for different neutron diffraction peaks and measured lattice strains indicated that observed hysteretic behavior cannot be attributed exclusively to movement of dislocation arranged in pileups as it was proposed earlier [14, 35], but rather to the bowing of dislocations arranged in dislocation walls most likely located in the hard grains. As the latter were probably formed by kinking of the hard grains, they were referred to as low angle kink boundaries (LAKB) [44].

The objective of this work is to further investigate to what extent the observed hysteretic behavior of Ti_2AlC can be attributed to the development of such a complex stress state (i.e. incompatibility stresses), microcracking, dislocation glide, and/or incipient kinking. In this study, high purity Ti_2AlC samples, with fine and coarse **grained** microstructures, were exposed to cyclic compressive loading, complemented with subsequent resonant ultrasound spectroscopy (RUS) tests and electron backscatter diffraction (EBSD) analysis in an attempt to shed more light on the room temperature hysteretic stress-strain behavior of the MAX phases. By performing small incremental steps in the applied cyclic stress and monitoring the changes in the residual (plastic) strain, area of hysteresis loops, elastic properties, and microstructure, four regions with distinct underlying mechanisms were identified.

2. EXPERIMENTAL PROCEDURE

High purity Ti_2AlC samples were prepared via a two-step processing in which Ti_2AlC powders were synthesized and subsequently densified by pulsed electric current sintering (PECS)². For the Ti_2AlC powder synthesis, elemental powders of Ti (99.5%, -325 mesh), Al (99.5%, -325 mesh) and TiC (99.5%, 2 μm), all from Alpha Aesar, USA, were mixed with molar ratio of $\text{Ti}:\text{Al}:\text{TiC} = 1.00:1.05:0.95$ optimized to achieve high purity powders, by ball milling for 24 hrs. The mixture was placed in alumina boats and pressureless sintered in a tube furnace (GSL1600X, MTI Corporation, USA) at 1400°C under UHP argon. The sintered compact of reacted powder was then drill milled and subsequently sieved to produce -170 mesh Ti_2AlC powders. **This powder was** then densified by PECS for 45 minutes at 1300°C under UHP Argon. In order to study the effect of grain size, a coarse grain sample was prepared following the previous steps but with an additional heat treatment of 24 hours at 1300°C in the tube furnace operated under argon atmosphere to promote grain growth.

The density of the sintered samples was measured by the alcohol immersion method (200 proof ethanol) based on Archimedes' principle, using the procedure that is described in more detail elsewhere [45, 46]. The microstructure and phase composition of the sintered

² This method is commonly but inaccurately referred to as Spark Plasma Sintering (SPS)

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4 samples were characterized by field emission SEM (Quanta 600 FEG, FEI, USA) equipped with a
5 back-scattered electron (BSE) detector and energy dispersive spectroscopy (EDS) system
6 (Oxford Instruments, UK), using both polished and polished and etched surfaces. The sample's
7 grain size and the volume percent of intermetallic impurity in the samples were determined
8 from SEM images using Image J software. X-ray diffraction (XRD) spectra were collected using a
9 diffractometer (D8 Discover, Bruker, USA) with Cu K α radiation at 40 kV and 40 mA, to
10 complement phase identification and quantification. The spectra were acquired in 8°– 80° 2 θ
11 range with 0.024° step and a rate of 3.5 °/min.
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15 In addition, EBSD analysis was used to characterize the microstructure of the as-sintered
16 and selected samples after exposure to compressive cyclic loading (see below). These samples
17 were mounted into 25 mm diameter epoxy blocks and mechanically polished using SiC grinding
18 papers and diamond suspensions with the final polishing using 60 nm OP-S colloidal silica for 15
19 minutes in order to provide a very smooth and flat surfaces. The epoxy blocks holding the
20 samples were then coated with silver paint in order to provide the necessary electron
21 grounding during the EBSD process. These samples were placed in a pre-tilted 70° specimen
22 holder to provide the high tilt angle required for EBSD. The EBSD scans were carried out using a
23 Zeiss Ultra field emission gun SEM equipped with Oxford Instruments AZtec integrated EDS and
24 EBSD system, with X-Max 20mm² silicon drift EDS detector and Nordlys-nano EBSD detector.
25 The scans were done with a step size of 0.2 μ m within a scanned area of 250 μ m x 250 μ m for
26 all the samples. All the scans were done with an accelerating voltage of 20 KeV at either 4x4 or
27 8x8 binning. Data acquisition and analysis were done using Oxford Instrument's AZTechKL and
28 HKL Tango software.
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32 Uniaxial cyclic compression tests were performed on cylindrical samples with
33 dimensions of 9 mm in diameter and 17 mm in length, cut by wire electron discharge machining
34 (Wire-EDM) and loaded in a servo-hydraulic testing machine (MTS-810, MTS, USA) at a
35 frequency of 0.5 Hz. At each testing step, the sample was cyclically loaded and unloaded twice
36 to the same magnitude of the maximum stress while an axial extensometer (632.13E-23, MTS,
37 USA), was attached directly on the sample to measure the deformation. After each testing step,
38 the sample was taken from the loading rig for RUS and placed back to the rig for the next
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testing step. In each subsequent testing step, the magnitude of the maximum stress was augmented by a small increment of 50 MPa. Those steps were repeated until sample failure occurred. The sample's elastic constants were measured with RUS (Quasar RUSpec, Magnaflux, USA) using the procedure described elsewhere [19]. RUS was chosen for its superb ability to detect defects in stiff solids independent of their location in the sample and its superior precision and repeatability in the determination of elastic constants [47, 48]. Note that due to the polycrystalline nature of the samples and since the sample size was much larger than the grain size, elastic isotropy was assumed in the calculation of elastic moduli. RUS spectra were collected prior to mechanical testing and after each increase in stress amplitude.

3. RESULTS AND DISCUSSION

3.1. Microstructural characterization of as-processed samples

Backscatter SEM micrographs of polished FG and CG samples in **Figure 3a** and **Figure 3b** illustrate uniform composition of the samples, with some porosity and small amounts of two other phases, identified by EDS to be Al_2O_3 (black areas in **Figure 3a** and **Figure 3b**) and TiAl_x (medium gray areas in **Figure 3a** and **Figure 3b**). The latter makes up about 3 vol % as determined by image analysis of BSE SEM images.

XRD results (not shown here) also confirmed the high purity of the processed samples, i.e. no peaks belonging to other phases but Ti_2AlC were observed. Moreover, EBSD analysis of as-processed samples (not shown) in different locations showed that all samples contained less than 3.3 vol% of Al_2O_3 , 1.4 vol% TiAl , and 0.16 vol% TiC impurities. Note that the porosity observed in the micrographs is in good agreement with the measured relative density of 98%. From the etched micrographs (**Figure 3c**) and by measuring sizes of more than 100 grains, the average grain diameter and thickness for the FG samples were determined to be $6 \pm 3 \mu\text{m}$ and $5 \pm 2 \mu\text{m}$, respectively. For the CG samples (**Figure 3d**), the average grain diameter of $17 \mu\text{m} \pm 10 \mu\text{m}$ and thickness of $8 \mu\text{m} \pm 3 \mu\text{m}$ was determined using the same procedure. The fine grain size microstructure with the low aspect ratio was chosen for this study to limit kinking to high stresses.

3.2. Cyclic compressive testing

Selected but typical stress-strain responses for both FG and CG Ti_2AlC samples, shown in **Figure 2**, resemble that of MAX phases reported elsewhere whereby the first load-unload cycle, or the first loop, exhibits some irrecoverable deformation or plastic strain, while the second load-unload loop is closed at lower stresses, or nearly closed at higher stresses [17]. Two quantities were extracted from the stress-strain curves at each maximum applied stress: (i) W_d from second load-unload cycle; and (ii) the first loop irrecoverable plastic strain ($\Delta\epsilon_{PL}$) otherwise commonly referred to as the opening of the first loop. Note that $\Delta\epsilon_{PL}$ determined in each subsequent testing step corresponds only to the irrecoverable strain produced by loading to the stress that is 50 MPa larger than that in the previous testing step. In addition, non-linear elastic strain (ϵ_{NL}) was determined after every second loading-unloading loop following procedure described elsewhere [20, 21]. The relative Young's modulus, i.e. modulus after cyclic loading divided by the modulus of the untested sample, was determined from the RUS results after each testing step, to quantify the changes of the elastic properties as these will indicate eventual damage accumulation in the sample.

The results for uniaxial compression testing in conjunction with RUS are summarized on a single plot for each microstructure, **Figure 4a** for the FG and **Figure 4b** for CG. These plots show W_d , first loop irrecoverable strain due to step increase of 50 MPa denoted as $\Delta\epsilon_{PL}$, and relative Young's modulus as a function of σ^2 . Results in **Figure 4** clearly indicate four different stress regions, further denoted as region I – IV, with substantially different mechanical responses. The threshold stresses between different regions are denoted by dashed lines in **Figure 4** and summarized in **Table 1**, while the underlying mechanisms in each region are discussed in more detail below.

Region I

In this stress region (FG: 0 to 175 MPa; CG: 0 to 100 MPa), Ti_2AlC exhibits a linear elastic behavior, without any detectable hysteresis. Consequently, no energy dissipation is detected. One FG sample exhibited its first hysteresis at 150 MPa and the second one at 200 MPa and

thus the average value of 175 MPa was chosen as the upper bound of region I. Similarly, the CG sample first exhibited hysteresis at an average stress of 100 MPa. Note that the irrecoverable strain and relative Young's Modulus do not change in this stress range, **Figure 4**. Therefore, we can conclude that the macroscopic mechanical behavior of Ti_2AlC is linear elastic in region I.

Region II

This stress region (FG: from 175 to 350 MPa; CG from 100 to 200 MPa) is characterized by a gradual increase in W_d per loading cycle, in conjunction with a steep increase in irrecoverable strain in the first loop with increasing maximum applied stress, and no observable change in elastic moduli, **Figure 4**. Note that the change of the first loop irrecoverable strain with stress shows more gradual progression with increasing stress (i.e. a change in slope is observed) above 350 MPa for FG, and above 200 MPa for CG. Therefore, those stresses were defined as transition stresses between regions II and III, $\sigma_{\text{II-III}}$. The lower transition stresses for region II ($\sigma_{\text{I-II}}$) are much lower than the threshold stresses of $\sigma_t = 379$ MPa for FG and $\sigma_t = 218$ MPa for CG, predicted using the KNE model (**Table 1**) [17]. Note that at stresses levels below the σ_t , the KNE model does not predict any hysteretic behavior or energy dissipation [21, 23, 26], which is in contradiction with the results presented here. However, the upper transition stress for region II happens to be very close to the σ_t predicted by IKB model. Since IKBs are not expected below σ_t it is reasonable to assume that the hysteretic behavior and small values of W_d per cycle observed in this region can be attributed to the development of complex incompatibility stresses state as a result of elastic/plastic anisotropy, and reversible glide of basal plane dislocations in soft grains, as it is suggested in the RF model [35]. This conclusion can be further supported by the fact that the irrecoverable strain measured during the first load-unload cycle increases rapidly with increasing maximum **loading** stresses. Note that loading within this stress region does not result in any damage since the Young's modulus does not change. The latter, completely rules out microcracking as the underlying mechanism responsible for the observed opening of the first loop and hysteretic stress-strain behavior. Therefore, it is reasonable to conclude that stresses in this region are sufficient to cause small hysteresis through reversible dislocation glide of basal plane dislocations in soft grains.

Region III

Mechanical response of Ti_2AlC in this region (FG: from 350 to 1100 MPa; CG from 200 to 650 MPa) is characterized by a steeper increase of W_d but a more gradual increase of $\Delta\epsilon_{PL}$ with increasing maximum compressive stress than in region II, **Figure 4**. Energy dissipated per cycle seems to increase linearly with σ^2 in this region, as it is indicated by the best fit line on the W_d vs. σ^2 plot in **Figure 4**. This result is in good agreement with previous experimental results and predictions of KNE model [16, 21, 23, 26], and challenges the RF model that predicts a linear increase in W_d with increasing σ [35]. However, further analysis of the results summarized in **Figure 5a**, actually shows that a slightly better fitting can be achieved by assuming that W_d is a function of both σ^2 and σ (i.e. assuming that $W_d = k + k' \cdot \sigma + k'' \cdot \sigma^2$), than only σ^2 (i.e. assuming that $W_d = k + k'' \cdot \sigma^2$) as suggested by the KNE model. This finding, together with the fact that $\Delta\epsilon_{PL}$ in region III increases much more slowly with increasing stress than in region II, suggest that some other deformation mechanism(s), beside reversible dislocation glide in soft grains as proposed in the RF model, must be operative and is (are) responsible for the observed hysteretic stress-strain behavior. However, W_d vs. ϵ_{NL} plots in **Figure 5b** still show linear trends for both CG and FG samples, as it is predicted by KNE model [20, 21, 25, 26]. From the slopes of the lines in **Figure 5b**, a magnitudes of Ω/b (where Ω is the energy dissipated by a dislocation loop unit length sweeping a unit area of according to the KNE model and b is Burgers vector) of 68.2-75.2 MPa and 35.6-36.4 MPa can be calculated for both fine and coarse grain structures, respectively, using KNE model and procedure described in more details elsewhere [20, 21, 25]. Those values are significantly larger than 24 MPa previously reported for fully dense Ti_2AlC with a grain diameter of $113 \pm 60 \mu\text{m}$ and the thickness of $14 \pm 7 \mu\text{m}$ [20] because samples used in this study had much smaller grain sizes. Similar effect of grain size on the magnitude Ω/b has been also reported before for another MAX phase, namely Ti_3SiC_2 [20, 21]. Such as strong dependence of Ω/b on the grain size, and thus on the size of eventual IKBs challenges previous conclusion that that Ω/b is identical to the critical resolved shear stress (CRSS) for dislocations gliding along the basal planes and thus direct measure of the frictional lattice stresses in the basal planes [20].

Note that whatever deformation mechanism(s) is (are) operative in region III, it (they) must result in cyclic strain hardening during loading because $\Delta\epsilon_{PL}$ increases with a much slower pace with increasing stress than in region II, but W_d is much larger. To further elucidate the underlying mechanisms in this region, the evolution of the microstructure was studied using EBSD and is discussed in more detail in the next section.

Last but not least, possible formation of microcracks in this region has to be discussed in more detail. The relative Young's modulus determined using RUS decrease monotonically with increasing stress, **Figure 4**. However, this decrease is very small, i.e. around 0.1% for FG after loading to 1100 MPa and 0.2 % for CG structure after loading to 650 MPa, and it approaches the sensitivity limits of RUS that has been reported to be around 0.17% [48]. In addition, results in **Figure 6** show that that decreases in the FG structure does not exceed 0.02 % after 100 cycles up to 700 MPa. Although, these results cannot completely rule out the formation of microcracks whose size and/or amount do not need to be large to affect significantly the elastic modulus, it is reasonable to conclude here that their opening and closing during cycling loading cannot be the dominant hysteretic mechanism as suggested in the MC model. In addition, the fact that cyclic hardening, rather than softening, occurs in this stress region (note that $\Delta\epsilon_{PL}$ in this region increases much more slowly with σ than in region II) additionally supports this conclusion since the formation of microcracks during loading would result in a much steeper increases in $\Delta\epsilon_{PL}$, i.e. cyclic softening, with increasing stress in this region when compared to region II.

Region IV

This stress region (FG: from 1100 MPa to failure at ≈ 1200 MPa, CG from 600 MPa to failure at ≈ 710 MPa) is characterized by a significant increase in W_d with increasing stress, exceeding the values predicted by the KNE model that are denoted by dotted lines on the bottom plots in **Figure 4**. More importantly, a steep decrease of elastic modulus, **Figure 4**, and even slight opening of the second hysteresis loop can be observed in this region (not shown here). These results suggest that microcracks start to form above the σ_{III-IV} , the transition stress,

producing a sudden and pronounced drop in relative elastic moduli and slight opening of the second loops. At these high stresses, the dissipation due to friction between microcracks faces also adds to the dissipation mechanism from region III thereby increasing the overall W_d dissipated in each loading cycle as predicted by the MC model. It is worth noting here that Poon et al. also reported [32] accumulation of microcracks at stresses of 540 MPa in the samples having slightly higher grain size than the CG samples in this study. This critical stress value is quite close to the value of σ_{III-IV} , 650 MPa, reported here for the CG structure. Last but not least, it is important to mention that the FG sample failed at 1.2 GPa, which is the highest compressive strength ever reported for Ti_2AlC .

3.3. EBSD Results

To grasp a better understanding of underlying hysteretic mechanism in region III, and to specifically explore the reason behind the cyclic hardening observed in that region, EBSD analysis was performed on FG and CG samples cycled twice to 500 MPa and compared to data acquired on as-sintered samples. EBSD is a valuable tool in analyzing evolution of deformation in polycrystalline MAX phases [49] because KBs and DWs result in changes in crystallographic orientation (misorientation) within individual grains. Therefore, the latter two features can be easily identified by analyzing misorientations on inverse pole figure (IPF) maps obtained by EBSD. Note that any eventual basal plane slip cannot be detected in EBSD results since it does not produced in any major misorientation within individual grains.

Figures 7 and 8 summarize the EBSD results for FG and CG samples, respectively, in the as-sintered condition (top rows) and for samples cycled to 500 MPa (bottom rows). The IPF maps of as processed samples in **Figures 7a and 8a**, show that both FG and CG samples have almost random texture with a weak alignment of the basal planes perpendicular to the loading direction applied during processing, as it is indicated by larger amount of red grains especially in the FG structure. Comparison of IPF maps before and after loading (**Figure 7a and 7b** for the FG structure and **Figure 8a and 8b** for the CG structure) show clearly that the microstructure evolves during loading, i.e. the color in each individual grain becomes less uniform after deformation. The latter suggests development of crystallographic misorientation in individual

grains during cyclic compressive loading. To further analyze misorientation development in each grain, the low angle grain boundaries (LAGBs) resulting in misorientation angles between 2° to 10° are plotted as red lines in **Figures 7c, 7d, 8c and 8d**, together with black lines that denote high angle grain boundaries (HAGBs) with misorientation angles exceeding 10° . Comparison of those results reveals much larger number of LAGBs in FG and CG samples cyclically loaded to 500 MPa than in as-sintered samples.

To further analyze if some of above mentioned microstructural features form predominantly in some grains because of their preferential orientation relative to the applied load, LAGBs and HAGBs are plotted over Schmid factor maps in **Figures 7e, 7f, 8e, and 8f**. Schmid factors on those maps were determined for easy slip (0001) $[11\bar{2}0]$ and therefore light blue grains denote longitudinal and transverse hard grains having Schmid factor close to zero, while yellow grains denote soft grains with Schmid factor close to 0.5. Careful inspection of these results shows that LAGBs form in all grains independent of their Schmid factor. At the same time, some of the grains are free of any LAGBs, independent of their orientation relative to the applied load. This suggests that deformation of each individual grain is governed not only by its orientation relative to direction of applied load, but also by orientation of all neighboring grains and thus incompatibility stresses that develop due to their plastic anisotropy. The latter, cannot be overemphasized because it demonstrates the importance of incompatibility stresses that develop during loading in region III on the overall mechanical response of the MAX phases. In addition, this finding challenges models used to capture the mechanical response of the MAX phases, such as EPSC, that are based on homogeneous inclusion method in which mechanical response of grains surrounding each individual grain is homogenized. Although, more work is needed to fully understand the evolution of inhomogeneous evolution of LAGBs in polycrystalline MAX phases during loading at room temperature, it is worth noting that those features seem to form preferentially, but not exclusively, in grains with middle to high Schmid factors (0.25-0.5). The latter is in good agreement with recently published results of in-situ neutron diffraction studies during cycling loading of Ti_2AlN that demonstrate plastic deformation of the grains in which the angle between the [0001] direction and the direction of applied stress is below approximately 70° [14].

A closer look at the geometry of LAGBs in **Figures 7 and 8** also reveals that they tend to form three different configurations. **Figure 9** shows select, but typical, configurations of LAGBs in grains denoted as A, B, C, D and E in **Figures 7e and 8e**, together with their Schmid maps and misorientation angle profiles along LAGBs. Observed LAGB configurations can be categorized as:

- (i) straight lines that span from grain boundary to grain boundary through the grain, **Figure 9b and Figure 9c**;
- (ii) closed loops in the middle of grains, **Figure 9c, 9d and 9e**; and
- (iii) open loops (curved lines) that start at grain boundaries and curl into the grains, **Figure 9a**.

Assuming that LAGBs consist of dislocation walls, the distance between dislocations can be expressed as $D = \frac{b/2}{\sin(\theta/2)}$ where the Burgers vector $b = \frac{1}{3}\langle 11\bar{2}0 \rangle = 3.05 \text{ \AA}$ [3]. If one basal dislocation is present in every Al basal plane, where $D = d_{\text{Al-Al}} = 6.8 \text{ \AA}$, then misorientation angle is 12.96° . Given that the misorientation angles across LAGBs range from 3° to 6° (see **Figure 8**) it follows that the observed LAGBs consist of dislocation walls with one dislocation located in every 4th to 9th Al plane. This, together with the fact that straight LAGBs span from grain boundary to grain boundary through the grains as it was observed and reported earlier in deformed MAX phases in the large numbers of post mortem studies [3, 4], suggest that observed LAGBs are nothing else but low angle kink boundaries (LAKB) as they are associated with deformation of individual grains by kinking and they confine KBs. Therefore, results presented here are the first direct evidence of LAKB formation in the MAX phases during cyclic loading even at relatively low stresses that are well below failure stresses, as it was recently predicted from the results of *in situ* neutron diffraction studies during cyclic loading of highly textured Ti_2AlC [44]. Note that not all parallel sets of straight LAGBs confine KBs, as it is illustrated in **Figure 9c**, because those walls consist of dislocations of the same sign. Therefore, it is reasonable to conclude that straight dislocation walls do not form exclusively by growth of IKBs to fully developed KBs, and thus not all of them are LAKB. It follows that another mechanism needs to be invoked to explain the presence of the walls consisting of dislocations of the same sign than that proposed by Orowan [9] and Frank and Stroh [10]. One possibility is

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4 simple bending of individual grains caused by anisotropic plastic deformation of surrounding
5 grains.
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8 The formation of closed and open LAGB loops, such as those in Figures 9a, 9c, 9d, and
9 9e in deformed MAX phases have never been reported before. Those loops resemble the
10 configuration of IKBs and partially dissociated IKBs, respectively, as predicted by KNE model [16,
11 17] since misorientation angle change across those loops indicates that their opposite ends
12 consist of dislocation walls of opposite signs. However, in contrast to IKBs, LAGB loops observed
13 here have relatively large diameter, exceeding in some cases 5 μm . Therefore they can be
14 referred to as LAKB loops, that did not fully developed into the straight LAKB. Note that those
15 loops form in all grains, not only in longitudinal hard grains, and that they do not completely
16 collapse upon unloading, as it is proposed by KNE model. Although more work is needed to fully
17 understand structure of LAKB loops and mechanisms that lead to their formation, it is
18 reasonable to conclude that incompatibility stresses are responsible for their formation during
19 loading in all grains independent of their orientation relative to applied load. In addition, those
20 stresses most likely prevent their total collapse or annihilation during unloading due to
21 attractive force between two dislocation walls of the opposite signs and/or their size is large
22 enough for their collapse upon unloading.
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37 Herein, the difference between the observed evolution of the microstructure in FG and
38 CG samples during compressive loading (Figures 7 and 8) has to be discussed in more detail.
39 When doing that, one has to consider that both microstructures were analyzed after cyclic
40 loading to 500 MPa, which is in the middle of stress region III for the FG structure and closer to
41 the upper limit for stress region III in the CG structure. Comparison of Figures 7d and 8d shows
42 that the amount of LAKBs is much higher in the CG structure than in the FG one. For example, a
43 large number of straight loops and can be seen in single grains in the CG structure, as
44 illustrated in the longitudinal hard grain circled with a dashed line in Figures 8b and 8f. This is
45 not surprising because the CG structure shows smaller resistance not only to dislocation glide in
46 basal planes (Hall–Petch effect) but also to the formation of KBs [3, 17, 21], when compared to
47 the FG structure. While the former would result in higher incompatibility stresses in the CG
48 structure due to easier formation of dislocation pile-ups in soft grains, the later will result in
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4 easier formation of KBs. Also, as the number of KBs seems to be smaller in the FG structure,
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6 they appear to form mostly in grains close to soft orientations for reasons that are not entirely
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8 clear at this moment.
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10 Results of EBSD analysis also show that the number of grains (where a grain is defined as
11 the area surrounded by HAGBs) in the examined area of $250 \times 250 \mu\text{m}^2$ increases from 3,634 in
12 the as-processed to 4,021 in the cyclically loaded FG samples, and from 896 in the as-processed
13 to 3,958 in the cyclically loaded CG samples. While the slight increase in number of grains in the
14 FG structure can be attributed to statistical difference between two examined areas, the
15 significant increase in the CG structure is a strong evidence that grain refinement and
16 development of substructure take place during cyclic loading, even at room temperature and
17 for very small plastic strains. Careful inspection **Figure 8f**, suggest that subgrains form mostly in
18 grains with high Schmid factors, i.e. soft grains, as it is illustrated in **Figure 10**. Although more
19 work is needed to fully understand the mechanisms responsible for the substructure
20 development in those grains, the misorientation profiles across those subgrains resemble those
21 previously observed in deformed Ti_3SiC_2 grains by TEM (**Figure 10c**). Note that the combination
22 of kinking and delamination in **Figure 10c** results in misorientation of up to approximately 30° ,
23 similar to that shown in **Figure 10a** and the formation of subgrains (circled by dashed line in
24 **Figure 10c**) similar to those observed in the sample we analyzed (**Figure 10b**). This conclusion is
25 additionally supported by the fact that highly fragmented grains contain areas with poor EBSD
26 indexing (purple in **Figure 10b**) due to delamination and microcracking.
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43 In conclusion for this section, EBSD results showing complex microstructural
44 development during room temperature cyclic loading have to be put in perspective with
45 mechanical test results. Observed refinement of microstructure due to the accumulation of
46 **LAGBs** and further formation of subgrains are in good agreement with observed cyclic
47 hardening in region III (i.e. much more gradual increase in $\Delta\epsilon_{\text{PL}}$ with increasing stress than in
48 region II) as those microstructural features are effective barriers for dislocation glide. However,
49 since energy dissipated per loading cycle in this stress region increases rapidly with increasing
50 stress, reversible glide of dislocation to and from pile-ups cannot be a major dissipation
51 mechanism as proposed by the RF model [35], because it would be hindered effectively by
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LAGBs or HAGBs that form during the first loading cycle. This finding further confirms recent conclusion of Shamma et al. [44] that hysteretic behavior in the stress region III cannot be attributed solely to the reversible dislocation glide. Taken into account excessive formation LAGBs during loading in the stress region III, majority of which are straight LAKBs or LAKB loops, it is reasonable to conclude that the major energy dissipation mechanism in region III is reversible movement (or bowing) of dislocations arranged in those boundaries --- not much unlike that proposed recently by Shamma et al. [44]. In addition, results presented here show that LAKBs appear in all grains independent on their orientation and that some of LAKB loops resembling IKBs are stable and do not collapse after unloading. Both those findings can be attributed to the development of complex stress states due to generations of local incompatibility stresses as a result of the high plastic anisotropy of MAX phases.

4. CONCLUSIONS

The results of uniaxial cyclic loading in compression, together with results of elastic modulus measurements and EBSD show that the room temperature mechanical response of both fine (FG) and coarse (CG) grain Ti_2AlC is quite complex and can be separated in the following four different stress regions (region I – IV) with substantially different underlining deformation mechanisms:

- **Region I** (FG: 0 to 175 MPa; CG: 0 to 100 MPa) is characterized as completely linear elastic behavior;
- **Region II** (FG: from 175 to 350 MPa; CG from 100 to 200 MPa) displays a hysteric behavior that is shown by a slightly open hysteretic loop but only during the first loading-unloading cycle, and closed loops for any subsequent cycling to the same stress level. In this region, the W_d observed can be attributed to the development of complex incompatibility stresses as a result of elastic/plastic anisotropy, and reversible glide of basal plane dislocations in soft grains, as suggested by the reversible flow model [35].
- **Region III** (FG: from 350 to 1100 MPa; CG from 200 to 650 MPa) shows a qualitatively similar stress strain behavior as the one observed in region II, but with a much steeper increase in W_d and a more gradual increase in the irrecoverable strain or opening of the first

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4 loop than in the region II. EBSD results show complex developments of the microstructure
5 comprising of a large number of straight LAKBs that span from grain boundary to grain
6 boundary through the grain, and closed or open LAKB loops. Both of them lead to cyclic
7 hardening. The reversible movement (or bowing) of those the boundaries --- not much
8 unlike what was proposed by the KNE model [17, 20, 21, 44]--- is proposed as a major
9 energy dissipation mechanism in this region. It is worth noting here that the EBSD results of
10 this study provide the first experimental evidence of the formation of closed LAKB in MAX
11 phases resembling IKBs.

- 12 • **Region IV** (FG: from 1100 MPa to failure, CG from 650 MPa to failure) stress strain behavior
13 as the ones observed in regions II and III, but is accompanied by a drop in the elastic
14 modulus due to damage (microcracks) formation. Friction at the microcracks' surfaces can
15 contribute to W_d , in addition to the other mechanisms mentioned above.

16 The results shown here highlight the fact that neither of the proposed models namely KNE,
17 microcracking or reversible flow model can fully account for the energy dissipated or describe
18 the complex mechanical behavior of the MAX phases over the entire range of stresses. This
19 comment notwithstanding, the majority of the energy dissipated and thus the most important
20 mechanism appears to be reversible movement of dislocation in LAKBs.

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FIGURE CAPTIONS

Figure 1: Schematic representation of different mechanisms proposed to explain hysteretic stress-strain behavior in the MAX phases. Green, red and blue lines denote orientation of basal plans in each grain. (a) Schematic of hypothetical microstructure consisting of soft (green), hard (red) and kinking (blue) grains; (b) Formation of IKB under the stress in kinking grain according to KNE model [16, 21, 23, 26]; (c) Stress induced delamination according to microcracking model [32]; (d) Dislocation glide in soft grains according to reversible flow model [35].

Figure 2: Stress-strain behavior of Ti_2AlC in compression at loading frequency of 0.5 Hz; (a) Fine grain (FG) structure and, (b) Coarse grain (CG) structure.

Figure 3: Backscatter SEM micrographs of: (a) polished FG Ti_2AlC sample sintered at 1300°C for 45 minutes, (b) polished CG Ti_2AlC sample sintered at 1300°C for 45 minutes with an additional heat treatment of 24 hr at 1300°C ; both micrographs show same small pores. Small dark areas of Al_2O_3 and grey areas of TiAl_x as identified by EDS; (c) and (d) are backscatter SEM images of etched FG and CG specimens, respectively, showing differences in grain sizes between the two microstructures.

Figure 4: Results for mechanical testing of (a) FG and, (b) CG. Bottom plots: W_d as a function of maximum applied stress squared; Middle plots: Irrecoverable strain from first load unload cycle as a function of maximum applied stress squared; Top plots: Relative Young's modulus as a function of maximum applied stress squared. Topmost scales indicate stress regions with different mechanical behavior observed in this study. Squared and circular symbols denote results obtained using two different samples for both microstructures.

Figure 5: W_d and per loading cycle plotted as (a) a function of maximum loading stress and (b) ϵ_{NL} in each cycle for FG (square symbols) and CG (round symbols) structure. Solid lines in (a) are the results of fitting assuming two different functions, namely $W_d = k + k'' \cdot \sigma^2$ (blue) and $W_d = k + k' \cdot \sigma + k'' \cdot \sigma^2$ (red), where k , k' and k'' are constants. Results are obtained using two different samples for each microstructure. Dashed lines in (b) are the results of fitting assuming that $W_d = A\epsilon_{NL} - C$, where A and C are constants.

Figure 6. Changes of the relative Young's modulus of the FG structure as a function of number of cycles up to 700 MPa.

Figure 7: EBSD results for FG as-sintered sample (top row) and FG cycled to 500 MPa (bottom row). Both compressive loads during sintering and cyclic loading were applied in the vertical direction. (a) and (b) are inverse pole figure (IPF) maps with the y-axis in the vertical direction; (c) and (d) Low (red lines) and high (black lines) angle grain boundaries maps; (e) and (f) Schmid factor maps with low (red lines) and high (black lines) angle grain boundaries.

Figure 8: EBSD results for CG as-sintered sample (top row) and CG cycled to 500 MPa (bottom row). Both compressive loads during sintering and cyclic loading were applied in the vertical direction. (a) and (b) are inverse pole figure (IPF) maps, with y axis in the vertical direction; (c) and (d) Low (red lines) and high (black lines) angle grain boundaries maps; (e) and (f) Schmid factor maps with low (red lines) and high (black lines) angle grain boundaries.

Figure 9: Misorientation profiles and unit cell orientations with respect to the vertically applied load across LAGBs or DWs in (a) grain A, (b) grain B, (c) grain C in **Figure 7e** as well as (d) grain D and (e) grain E in **Figure 8e**. Inserts show magnified Schmid factor maps for corresponding grains with low (red lines) and high (black lines) angle grain boundaries.

Figure 10: Misorientation profile and unit cell orientations with respect to the vertically applied load across grain F in **Figure 8e**. (b) Magnified Schmid factor map for corresponding grain with low (red lines) and large (black lines) angle grain boundaries, and unindexed areas (purple). (c) TEM of kink and low angle kink boundaries in deformed Ti_3SiC_2 from [50].

TABLE CAPTIONS

Table 1: Transition stresses between regions I and II ($\sigma_{\text{I-II}}$), regions II and III ($\sigma_{\text{II-III}}$) and regions III and IV ($\sigma_{\text{III-IV}}$) determined from **Figure 4** for both FG and CG microstructures. * σ_t is threshold stress as defined in IKB model. It is calculated from the intercept of the W_d vs. σ^2 plot with σ^2 -axis as described in [17].

Figure 1: Schematic representation of different mechanisms proposed to explain hysteretic stress-strain behavior in the MAX phases. Green, red and blue lines denote orientation of basal plans in each grain. (a) Schematic of hypothetical microstructure consisting of soft (green), hard (red) and kinking (blue) grains; (b) Formation of IKB under the stress in kinking grain according to KNE model [16, 21, 23, 26]; (c) Stress induced delamination according to microcracking model [32]; (d) Dislocation glide in soft grains according to reversible flow model [35].

Figure 2: Stress-strain behavior of Ti_2AlC in compression at loading frequency of 0.5 Hz; (a) Fine grain (FG) structure and, (b) Coarse grain (CG) structure.

Figure 3: Backscatter SEM micrographs of: (a) polished FG Ti_2AlC sample sintered at 1300°C for 45 minutes, (b) polished CG Ti_2AlC sample sintered at 1300°C for 45 minutes with an additional heat treatment of 24 hr at 1300°C ; both micrographs show same small pores. Small dark areas of Al_2O_3 and grey areas of TiAl_x as identified by EDS; (c) and (d) are backscatter SEM images of etched FG and CG specimens, respectively, showing differences in grain sizes between the two microstructures.

Figure 4: Results for mechanical testing of (a) FG and, (b) CG. Bottom plots: W_d as a function of maximum applied stress squared; Middle plots: Irrecoverable strain from first load unload cycle as a function of maximum applied stress squared; Top plots: Relative Young's modulus as a function of maximum applied stress squared. Topmost scales indicate stress regions with different mechanical behavior observed in this study. Squared and circular symbols denote results obtained using two different samples for both microstructures.

Figure 5: W_d and per loading cycle plotted as (a) a function of maximum loading stress and (b) ϵ_{NL} in each cycle for FG (square symbols) and CG (round symbols) structure. Solid lines in (a) are the results of fitting assuming two different functions, namely $W_d = k + k'' \cdot \sigma^2$ (blue) and $W_d = k + k' \cdot \sigma + k'' \cdot \sigma^2$ (red), where k , k' and k'' are constants. Results are obtained using two different samples for each microstructure. Dashed lines in (b) are the results of fitting assuming that $W_d = A\epsilon_{NL} - C$, where A and C are constants.

Figure 6. Changes of the relative Young's modulus of the FG structure as a function of number of cycles up to 700 MPa.

Figure 7: EBSD results for FG as-sintered sample (top row) and FG cycled to 500 MPa (bottom row). Both compressive loads during sintering and cyclic loading were applied in the vertical direction. (a) and (b) are inverse pole figure (IPF) maps with the y-axis in the vertical direction; (c) and (d) Low (red lines) and high (black lines) angle grain boundaries maps; (e) and (f) Schmid factor maps with low (red lines) and high (black lines) angle grain boundaries.

Figure 8: EBSD results for CG as-sintered sample (top row) and CG cycled to 500 MPa (bottom row). Both compressive loads during sintering and cyclic loading were applied in the vertical direction. (a) and (b) are inverse pole figure (IPF) maps, with y axis in the vertical direction; (c) and (d) Low (red lines) and high (black lines) angle grain boundaries maps; (e) and (f) Schmid factor maps with low (red lines) and high (black lines) angle grain boundaries.

Figure 9: Misorientation profiles and unit cell orientations with respect to the vertically applied load across LAGBs or DWs in (a) grain A, (b) grain B, (c) grain C in **Figure 7e** as well as (d) grain D and (e) grain E in **Figure 8e**. Inserts show magnified Schmid factor maps for corresponding grains with low (red lines) and high (black lines) angle grain boundaries.

Figure 10: Misorientation profile and unit cell orientations with respect to the vertically applied load across grain F in **Figure 8e**. (b) Magnified Schmid factor map for corresponding grain with low (red lines) and large (black lines) angle grain boundaries, and unindexed areas (purple). (c) TEM of kink and low angle kink boundaries in deformed Ti_3SiC_2 from [49].

Sample	σ_{I-II} (MPa)	σ_{II-III} (MPa)	σ_{III-IV} (MPa)	σ_t^* (MPa)
FG	175	350	1100	379
CG	100	200	650	218

* σ_t is threshold stress as defined in IKB model. It is calculated from the intercept of the W_d vs. σ^2 plot with σ^2 -axis as described in ¹⁷.

Table 1: Transition stresses between regions I and II (σ_{I-II}), regions II and III (σ_{II-III}) and regions III and IV (σ_{III-IV}) determined from **Figure 4** for both FG and CG microstructures. * σ_t is threshold stress as defined in IKB model. It is calculated from the intercept of the W_d vs. σ^2 plot with σ^2 -axis as described in [17].

Graphical abstract:

Stress-strain behavior of Ti_2AlC in compression at loading frequency of 0.5 Hz; (left) Fine grain (FG) structure and, (right) Coarse grain (CG) structure.

Results for mechanical testing of (a) FG and, (b) CG. Bottom plots: W_d as a function of maximum applied stress squared; Middle plots: Irrecoverable strain from first load unload cycle as a function of maximum applied stress squared; Top plots: Relative Young's modulus as a function of maximum applied stress squared. Topmost scales indicate stress regions with different mechanical behavior observed in this study.

EBSD results for CG as-sintered sample (top row) and CG cycled to 500 MPa (bottom row). Both compressive loads during sintering and cyclic loading were applied in the vertical direction. (a) and (b) are inverse pole figure (IPF) maps, with y axis in the vertical direction; (c) and (d) Low (red lines) and high (black lines) angle grain boundaries maps; (e) and (f) Schmid factor maps with low (red lines) and high (black lines) angle grain boundaries. (red lines) and high (black lines) angle grain boundaries maps; (e) and (f) Schmid factor maps with low (red lines) and high (black lines) angle grain boundaries. These results illustrate microstructure development and formation of low angle grain boundaries during cyclic loading in compression at room temperature.