Title: Inelastic strain recovery of a dynamically deformed unidirectional Ag-Cu eutectic alloy

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Abstract:

In this work we report experimentally observed inelastic recovery within a bulk directionally-cast Ag-Cu eutectic system tailored to possess a high density of incoherent twin interfaces. Incremental high strain-rate compression experiments were performed along the [101] crystallographic direction. Initial loading to a compressive strain of 0.10 was followed by inelastic strain recovery of 0.02 to 0.035. Further incremental loading saw decreasing inelastic strain recovery with each additional loading increment. Once a total compressive strain of 0.22 was reached there was cessation of inelastic strain recovery. Quasi-static compression and thermal experiments were conducted to elucidate the necessary loading conditions for inelastic recovery to occur in the Ag-Cu material and to provide insight of possible active recovery mechanisms.

Keywords: Strain recovery; silver-copper; twin boundary; ultra-fine-grain material; eutectic.

Introduction:

Early work on the study of multi-layered metallic bi-phase composite material systems explored the relationship between layer thickness and yield strength [e.g., 1-3]. From these experimental studies it has been proposed that the dislocation processes change with decreasing layer thickness; transitioning from dislocation pile-up formation, to confined layer slip and finally when individual layer thicknesses are a few nanometers, to dislocations crossing the interface. A review of these mechanisms can be found elsewhere [4] as they will not be described here in detail. Recent work has moved beyond the initial studies which treated interfaces as phenomenological barriers, and instead looks to identify the effects of interface atomic structure on dislocation nucleation, dislocation transmission, and interface shear resistance [e.g., 5, 6, 7, 8, 9, 10].

The Cu-Nb material system has received significant attention because the interface structure can be tailored through using different processing routes. When produced using physical vapor deposition (PVD) atomically flat interfaces with the $Cu\{111\}\langle 110 \rangle \parallel Nb\{110\}\langle 111 \rangle$ Kurdjmov-Sachs (K-S) orientation relationship are obtained, where $\{ \}$ denotes the family of common interface planes and $\langle \rangle$ indicates the family of common interface plane normals [e.g., 5, 6]. Alternatively, if accumulative roll bonding (ARB) is used [7, 8, 9,10] the interface is stepped with regular facets [11]. It has been shown through molecular dynamics simulation that dislocation nucleation and transmission processes as well as interface shear strength are controlled by interface structure. In summary, the atomic structure of a bi-phase interface determines the conditions under which dislocation nucleation and transmission can occur [12, 13], the ability of an interface to act as a strong or weak barrier to dislocation motion [14] and interface shear resistance [13, 15, 16]. Similar to the Cu-Nb system, the atomic interface

structure in the Ag-Cu multilayered material system can be tailored during fabrication, and more specifically in this study through the solidification rate chosen during directional casting. Low cooling rates result in micron scale microstructured material with the cube-on-cube interface orientation ($Ag(\bar{1}11) \parallel Cu(\bar{1}11)$), with aligned slip systems across the bi-phase interface) [17, 18], while the highest cooling rates result in an ultrafine-grained microstructured material with a coherent twin interfaces [19], intermediate cooling rates result in incoherent twin interfaces predominantly occurring with ($Ag(\bar{3}13) \parallel Cu(\bar{1}12)$) habit planes, with a shared $(1\bar{1}\bar{1})$ plane) [17, 20, 18]. Previous studies have examined the cube-on-cube interface structure capturing anomalous twinning in Cu under conditions that would not normally exhibit twinning [16, 17, 21]. Deformation communication across the bi-phase interface occurs due to Ag, which twins readily, providing sufficient twinning partials at the interface which then are transmitted into Cu resulting in twinning deformation within the Cu phase [16]. Therefore, atomic interface structure plays a defining role in how materials with a high density of interfaces accommodate deformation at the nano-/ultrafine length scales which in turn effects the bulk material response.

A particularly interesting study by Qin et al. [22] has demonstrated a dislocation-twin boundary interaction that results in a complete recovery of plastic deformation. In their study a penta-twinned Ag nano-wire was examined through quasi-static tension experiments and molecular dynamics (MD) simulations. They propose that the internal stress field of the penta-twinned microstructure and the repulsive stress-field at the twin boundary can drive partial dislocations back to their nucleation sites providing a mechanism for inelastic recovery of plastic deformation. The study by Qin et al. [22] is just one of multiple recent studies to demonstrate inelastic recovery in an appropriately structured single or bi-phase face-centered cubic (FCC) structured material. Experiments by Rajagoapalan et al. [23, 24] saw inelastic recovery in PVD-

produced free-standing nanostructured Al and Au films which they attributed to thermally assisted processes and small grain sizes. Lonardelli et al. [25] observed substantial recovery, on the order of one-quarter of the applied 0.04 compressive strain, in a bi-modal grain size Al specimen. They attribute the observed inelastic strain recovery to the combination of large ultrafine (100-500 nm) grains and nanocrystalline (<100 nm) grains to generate inhomogeneous strains and high levels of residual stress. The residual stress provides the driving force to propel dislocations backwards within the ultrafine grains, demonstrating the process may not need be thermally assisted. And finally the simulations of Li and Chew [26], saw plastic recovery through the reversal (i.e., de-twinning) of deformation twins. They credit the coherency stresses between Cu and Ag at the interface as the driving factor for the observed twin reversal and associated inelastic recovery. From the above studies it is evident that there are a number of microstructural features and loading conditions that could provide the necessary conditions for inelastic recovery to occur.

Motivated by these previous studies demonstrating inelastic strain recovery in appropriately structured FCC material systems, we investigate the effect of a high density of $Ag(\bar{3}13) \parallel Cu(\bar{1}12)$ coherent twin interfaces in the Ag-Cu material system. Dynamic compression loading was conducted along the [101] growth direction (0°), at 45° to the growth direction, and perpendicular (90°) to the growth direction. The high strain-rate deformation response along the [101] direction (0°) saw strain recovery in excess of that which can be accounted for solely through elastic recovery, pointing to the presence of an inelastic recovery mechanism. To examine the necessary conditions of inelastic recovery in the Ag-Cu systems, further quasi-static and thermal experiments were conducted to support the results obtained from dynamic experiments in which inelastic recovery occurred. Based on the experimental evidence

reported herein as well as other evidence of inelastic recovery in literature, as outlined above, the mechanism(s) responsible for the observed inelastic recovery shown here in the Ag-Cu system are proposed.

Experimental Technique:

Two rods of unidirectional Ag₆₀Cu₄₀ (subscripts indicating atomic percent composition) eutectic alloy were produced using the directional solidification technique described in Eftink et al. [17, 20] and Kingstedt et al. [18] at a furnace removal rate of 73 mm/hr. The resulting microstructure is comprised of alternating layers of Ag and Cu, see Figure 1(a), which shows a backscattered electron image of the material viewed parallel to the [101] growth direction. In Figure 1(a) the thin dark layers are Cu and the lighter layers are Ag. A boundary between colonies has been traced to highlight that neighboring eutectic colonies solidify with a rotation with respect to each other. The nominal (average) bi-layer thickness of Ag and Cu is 527 nm, with Ag having nominal individual layer thickness of 373 nm and Cu having nominal individual layer thicknesses of 154 nm. Layer thicknesses were determined using the following line intercept method. Scanning electron microscopy (SEM) micrographs viewing perpendicular to the [101] growth direction, were greyscale thresholded to obtain contours matching the Ag-Cu bi-phase interfaces. Horizontal lines were placed over the contour image finding intersection points between the contours (i.e., Ag-Cu interfaces) and the overlaid horizontal lines. Ag and Cu layer thicknesses were determined from the intersection points. Measurement of the layer thicknesses are shown in Figure 1(b) from which the representative average thicknesses are presented.

The material system used in this study is identical to the nominally 500 nm bi-layer material used in the comprehensive study of unidirectional Ag-Cu under dynamic compression

conducted by Kingstedt et al. [18]. A singular [101] growth direction of the Cu and Ag phases across all colonies, with crystallographic rotations about the [101] growth direction along the length of the casting [20]. The bi-phase interfaces are incoherent twin with $Ag(\bar{3}13) \parallel Cu(\bar{1}12)$ habit planes with a shared $(1\bar{1}\bar{1})$ slip plane, and cube-on-cube with $Ag\{\bar{1}11\} \parallel Cu\{\bar{1}11\}$ habit planes with aligned slip systems across the interface [20]. The area fraction of the incoherent twin orientation relationship comprises more than 66% of the total interface area [20].

Specimens for dynamic loading were prepared such that the loading direction is 0° (coincidental), at 45° and 90° to the [101] growth direction. Specimens for quasi-static loading were prepared for loading along the [101] growth direction only. Specimens were machined using electro-discharge machining (EDM) to standard length-to-diameter ratios, one-half for dynamic testing [27] and two for quasi-static testing [28]. Specimens were nominally 8 mm in diameter when loading along the [101] direction, and 7.5 mm for the 45° and 90° loading directions. Specimen faces were polished and confirmed for planarity prior to testing. Dynamic loading was conducted on a 12.7 mm diameter compression Kolsky bar [29,30] at strain rates on the order of 1500 s^{-1} . The Kolsky bar apparatus and the associated one-dimensional wave propagation analysis procedure are well documented [e.g., 31, 32, 33] and for brevity will not be described here. Quasi-static loading was performed on a MTS servo-hydraulic load frame at a strain rate of 10^{-3} s^{-1} . Machine compliance was accounted for using the method outlined by Kalidindi et al. [34].

Results:

Dynamic mechanical response:

Under dynamic loading conditions specimens loaded along the [101] direction exhibited strain recovery upon unloading that is greater than can be attributed solely to elastic recovery, indicating that a secondary recovery mechanism (i.e., inelastic recovery) may be active. Loading at 45° and 90° to the [101] growth direction did not exhibit inelastic recovery. To illustrate, the compressive true stress-strain material response along each loading direction is shown in Figure 2. Two curves are shown for the 0° loading orientation (circle and square markers), one from each casting, and one curve for the 45° (down triangle) and 90° (up triangle) loading orientations. The nearly vertical dashed lines indicate the unload path for elastic strain recovery. The predicted elastic strain recovery (ε_E) is determined from the final stress-state at unloading divided by the specimen elastic modulus. Markers along the bottom abscissa indicate two different strain measurements. Filled markers indicate the final true strain state before unloading from one-dimensional analysis of the Kolsky bar strain gauge signals, henceforth referred to as dynamic strain (ε_{Dy}). The open markers indicate the true strain determined from physical measurement of specimens immediately after deformation, henceforth referred to as accumulated strain (ε_A). Note the markers used to identify each stress-strain curve correspond to those on the bottom abscissa to indicate dynamic (ε_{Dy}) and accumulated (ε_A) strain.

For the 0° loading orientation arrows are drawn from the predicted strain after elastic recovery (i.e., $\varepsilon_{Dy} - \varepsilon_E$) to the accumulated strain (ε_A) to clearly indicate that a significant amount of inelastic strain recovery ($\varepsilon_I = \varepsilon_{Dy} - (\varepsilon_E + \varepsilon_A)$) occurs in this loading orientation. The levels of inelastic recovery shown in Figure 2 for the curves shown are $\varepsilon_I = 0.036$ (curve with circle markers), and $\varepsilon_I = 0.018$ (curve with square markers). For the 90° loading orientation there is a discrepancy of 0.006 between the predicted strain after elastic recovery and

accumulated strain. Due to the small magnitude of this discrepancy, the inelastic recovery was deemed to be insignificant in the 90° loading orientation compared to the 0° loading orientation. In the 45° loading orientation the accumulated strain exceeds the dynamic strain ($\varepsilon_A > \varepsilon_{Dy}$) due to there being multiple loading passes from reflecting waves in the Kolsky pressure bar. The 45° orientation has the lowest strength of the three loading orientations and therefore requires the lowest stress to facilitate deformation from the reflecting waves. To summarize, inelastic recovery was unique to the 0° loading orientation. Furthermore inelastic recovery is unique to the 527 nm bi-layer unidirectional cast Ag-Cu eutectic material system whose bi-phase interfaces are predominantly $Ag(\bar{3}13) \parallel Cu(\bar{1}12)$ incoherent twin, as inelastic recovery was not observed in unidirectional cast Ag-Cu eutectic material systems with bi-layer thicknesses greater than 527 nm [18].

To examine the level of accumulated strain at which there is an exhaustion of inelastic strain recovery further, incremental loading of 0° orientation specimens was conducted, as shown in Figure 3. The dashed curve shown in Figure 3 is for a specimen subjected to a single loading pass and set aside for microstructural analysis; this curve is identical to the circular marked curve in Figure 2. The solid curves show the response of a single specimen subjected to incremental loading passes until exhaustion of inelastic recovery (i.e., $\varepsilon_A \geq (\varepsilon_{Dy} - \varepsilon_E)$). Similar to Figure 2 the markers along the bottom abscissa in Figure 3 indicate that dynamic strain (filled markers), and accumulated strain (open markers), for each loading increment. It can be seen that as the accumulated strain increases (moving to the right in Figure 3) there is a decrease in the separation between the dynamic strain and the accumulated strain (i.e., a reduction in the total recovered strain). After the final loading increment to a total accumulated strain of 0.22 there is a cessation in the inelastic recovery (i.e., $\varepsilon_I = 0$). A summary of the results obtained for single

loading pass and incrementally loaded 0° specimens, including stress-strain curve not shown (second casting specimen 2) are presented in Table 1, showing the dynamic strain (ε_{Dy}), accumulated strain, total recovered strain ($\varepsilon_R = \varepsilon_{Dy} - \varepsilon_A = \varepsilon_E + \varepsilon_I$), and the recovered elastic (ε_E) and inelastic strains (ε_I). Details of the temperature rise associated with the work of deformation are also included in Table 1 and will be discussed in following sections. Figure 4 provides a graphical representation of the inelastic strain recovered against the accumulated strain. It is apparent that as the accumulated strain increases there is a decrease in the recovered inelastic strain, up to 0.22 at which point there is a cessation of inelastic recovery. A similar cessation of inelastic recovery at strains of 0.25 has been observed in recent molecular dynamic simulations of a Ag-Cu system possessing incoherent twin interfaces [35].

Quasi-static mechanical response:

Quasi-static experiments were conducted to examine the propensity of inelastic recovery at low strain-rates (10^{-3} s^{-1}). The loading and unloading response of a compressively deformed specimen in increments of 0.03 strain, up to a total strain of 0.12 is shown in Figure 5. The slight gap between the loading and unloading portion of the stress-strain curves is due to load relaxation when reversing the cross-head direction. The solid line from the top abscissa indicates the accumulated specimen strain after unloading, indicating limited if any strain recovery can be captured by far-field measurements under quasi-static conditions. However, should recovery occur locally it may not be resolvable in the far-field load frame displacements. Therefore, during loading and unloading three-dimensional digital image correlation (3D-DIC) was conducted to capture specimen strain field maps. In 3-D DIC, stereo imaging of a speckle patterned surface is conducted with two synchronized cameras, see Figure 6(a) for a

representative speckle pattern. Using commercial software (Vic3D), three-dimensional surface maps of displacements and strains are obtained. Figure 6(b) shows the unloading stress-strain response and strain fields of the vertical strain component (i.e., the loading direction) at four points: at the onset of unloading (Figure 6(c)), two points during unloading (Figure 6(d) and 6(e)), and at a point after there is a visible gap between the specimen and cross-head (Figure 6(f)) to study the material in a macroscopically stress free condition. The color maps chosen span the entire range of strain in the vertical direction during unloading. The strain fields shown in Figure 6 highlight the uniformity of the vertical strain field during unloading, and also capture strains that match those predicted for elastic recovery only. Therefore, based on the quasi-static experiments and the uniform DIC strain fields with amplitudes matching those of elastic recovery, we can conclude that inelastic strain recovery does not occur under quasi-static loading conditions at room temperature. Due to inelastic recovery not occurring in the 45° or 90° loading orientations, quasi-static experiments were not conducted along those loading paths.

Response to thermal treatments:

In high-strain rate loading experiments such as those conducted with the Kolsky bar, adiabatic conditions can occur depending on specimen and Kolsky bar material, and the loading pulse duration [36, 37]. The dynamic experiments conducted in this study can be classified as adiabatic, and thus the temperature rise associated with plastic work remains in the specimen for the loading duration. If the temperature rise is significant it can play a role in the deformation response as has been observed in the formation of adiabatic shear bands [38], and therefore may be a factor in explaining why plastic strain recovery is seen dynamically (adiabatic conditions) but not quasi-statically (isothermal conditions).

The transient heat conduction equation is an additional field equation used in coupled thermomechanical problems such as the dynamic loading conducted in the current study. The transient heat equation provides a relationship between known mechanical fields and unknown temperature fields, given by

$$k\nabla^2 T + \beta \sigma_{ij} \dot{\varepsilon}_{ij}^p - \alpha T_0 \frac{E}{1-2\nu} \dot{\varepsilon}_{kk}^e = \rho c_p \dot{T}, \quad (1)$$

where, the over dot refers to a derivative with respect to time, σ_{ij} and ε_{ij} are the Cartesian stress and strain fields with superscripts e and p respectively indicating the elastic and plastic components, k is the thermal diffusivity, α is the coefficient of thermal expansion, c_p is the heat capacity, E is the elastic modulus, ν is the Poisson ratio, T_0 is the ambient temperature, T is the specimen temperature, and lastly β is the work-rate to heat-rate conversion factor. The first term of the transient heat equation can be neglected for the experiments reported here due to adiabatic conditions ($k\nabla^2 T = 0$). The third term is the thermo-elastic effect, a quantity that typically is on the order of fractions of a degree Celsius [39]. Due to the small contribution of the thermo-elastic effect on the final temperature state it is often neglected. With these assumptions, equation (1) can be re-written as follows

$$\beta = \frac{\rho c_p \dot{T}}{\sigma_{ij} \dot{\varepsilon}_{ij}^p}. \quad (2)$$

Studies by Mason et al. [36,40], Zehnder [41] and Aravas et al. [42] have shown that the work-rate to heat-rate conversion factor is material, strain, and strain-rate dependent having values ranging between 0.6 and 1.0. Alternatively, the work of Kapoor and Nemat-Nasser [37] propose a plastic work to heat conversion factor (η) that is not dependent on differentiation of discrete experimental data. Their conversion of plastic work to heat factor takes the form

$$\eta = \frac{\rho c_p \Delta T}{\int_0^\varepsilon \sigma d\varepsilon}, \quad (3)$$

where, ΔT is the change in temperature due to plastic work. Kapoor and Nemat-Nasser argue, based on experimental observations, that there is a full conversion of plastic work to heat when materials undergo deformation, $\eta = 1$ [37]. For the current study, *in-situ* experiments monitoring the temperature evolution with respect to deformation were not conducted due to limited material availability. As a conservative estimate of the final thermal state of deformed specimens it will assumed that there is a complete conversion of plastic work to heat ($\eta = 1$). Invoking equation (3) and assuming that the density and heat capacity are appropriately represented by temperature independent rule of mixtures estimates ($\rho_{ROM} = 9878 \text{ kg / m}^3$ and $c_{p \text{ ROM}} = 298 \text{ J / kg } ^\circ\text{C}$). The temperature rises predicted for all loading in this study were less than 12°C . A summary of the temperature rise for each loading increment can be found in Table 1.

In the quasi-statically loaded specimen, isothermal conditions prevail. Therefore, the specimens remain at, or very near, ambient temperature during loading. Previous studies of material systems demonstrating inelastic recovery [23,24] have shown inelastic recovery to be thermally assisted. Rajagaopalan et al. [23,24] proposed that the thermal energy provided through annealing treatments drives the recovery mechanism through wells in the energy landscape. While the temperature rise predicted for the deformed specimens is small compared to the annealing schedule of previous studies [23,24] the role of temperature cannot be ignored. Hence, to investigate the role that temperature plays on the inelastic recovery response quasi-statically deformed specimens were sectioned into two pieces. One piece was set aside for microstructural analysis and the other was placed in a furnace to achieve a thermal state mimicking those of the dynamically deformed specimens that demonstrated inelastic recovery.

Thermal treatments resulting in temperature rises similar to those provided in Table 1 did not observe recovery. Further heat treatments to 100 °C for 30 minutes also did not result in observable recovery.

Microscopy analysis was conducted on quasi-static, quasi-static thermally treated and dynamically deformed specimens at multiple length scales using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). SEM micrographs show co-deformation of both the Ag and Cu phase [18]. A detailed TEM study of dynamically deformed material identical to that of this study can be found in Eftink et al. [20]. TEM analysis from dynamically deformed specimens found deformation in the form of localized dislocations in Cu, and dislocation cells in Ag. An additional molecular dynamics and microscopy study can be found in the work of Eftink et al. [35]. They report a number of interesting features including rotation of the incoherent twin interface due to deformation, decreases in dislocation density upon unloading, and dislocation tangles in quasi-statically loaded specimens that did not exhibit inelastic recovery [35].

Discussion

Based on the experimental evidence presented here, it is proposed that the mechanism(s) to facilitate inelastic recovery are not temperature sensitive for the unidirectional Ag₆₀Cu₄₀ material system, and instead are too complex to be isolated to a single mechanism from the experimental evidence shown here. It is acknowledged that the thermal aspects of the dynamic loading may aid driving mechanism(s) through wells in the energy landscape towards recovery, but should they occur, they play a small role if any due to the low temperature rise predicted even with a complete conversion of plastic work to heat ($\eta = 1$). This proposal is reinforced by

the time scale over which the inelastic recovery occurs. Thermally activated diffusion and grain boundary sliding act at slow rates, on the order of 10^{-7} to 10^{-9} s⁻¹, whereas inelastic strain recovery was seen in measurements made within seconds of the completion of dynamic loading.

We propose possible mechanisms that could aid in inelastic recovery within the examined material system based on microscopy observations of the Ag-Cu material system [20, 35], experimental evidence of recovery in FCC materials [22,23,24,25] and modeling efforts [22,26, 35] results. The three features proposed to contribute to the inelastic recovery in the dynamically deformed Ag-Cu material system are: 1) the high density of incoherent twin boundaries within the microstructure and the associated stress-fields at the bi-phase interface, because inelastic recovery was only observed in the Ag-Cu material system with a high density of the $Ag(\bar{3}13) \parallel Cu(\bar{1}12)$ incoherent twin orientation relationship, 2) the internally heterogeneous stress-state that develops during deformation due to the comparative size difference of the Cu and Ag layers, which could act to help drive dislocations along paths resulting in recovery, and 3) loading rate because the inelastic recovery was only observed under dynamic loading conditions.

Detailed microscopy of the Ag-Cu system conducted by Eftink et al. [20] captured a rotation of the interface post-deformation in both dynamically and quasi-statically deformed specimens. Prior to deformation the incoherent twin interface orientation has a $Ag(\bar{3}13) \parallel Cu(\bar{1}12)$ habit plane, with a shared $(1\bar{1}\bar{1})$ twin plane. Under accumulated strains of 0.05 and 0.22 the misorientation of the incoherent twin interface, measured by the angle between the shared $(1\bar{1}\bar{1})$ plane about the $[101]$ growth direction, increases from 2.0° to between 5.9° and 14.0° [20]. The change in interface structure is a result of the lack of deformation communication between Ag and Cu through the twin interface [20]. Therefore, the incoherent

twin interface acts as a strong boundary to dislocation motion which likely leads to the development of a heterogeneous stress-state between layers.

Molecular dynamics simulations of a Ag penta-twinned material [22] showed that partial dislocation loops nucleated at free surfaces, were unable to communicate across the twin boundaries, and that they retracted back to the free surface during unloading. It was claimed that the repulsive force of the twin boundaries on the partial dislocations [43,44] reversed the motion of dislocations by pushing dislocations backwards towards their nucleation sites. There will also be an energetic reduction by clearing the stacking fault caused by the partial dislocation that will drive the partial dislocation loop back to the free surface. Alternatively, the simulations by Li and Chew [26] of a lamellar Ag-Cu with equal thickness layers of 2 nm or 8 nm found retraction of twins back to their nucleation sites to recover plastic strain during unloading at stress below 1 GPa. In their simulations the twin retraction is driven by alternating in-plane tensile compressive residual stresses due to coherency stresses caused by lattice misfit at the bi-phase interface [26].

Recent molecular dynamics simulations of the response of a Ag-Cu material with the incoherent twin interface structure revealed that the rotation of the interface under loading provides a local stress that drives dislocation activity at the interface [35]. From the simulations, illustrative evidence of the retraction mechanisms that occur are presented providing support of the proposal that the high density of incoherent twin interfaces is necessary to facilitate the conditions necessary for inelastic recovery in the Ag-Cu system [35].

HRTEM characterization of the incoherent twin interface captured a random distribution of dislocations at the interface [20]. As the length scale of nanocomposites is reduced a greater portion of lattice dislocations sites occur at interfaces opposed to within individual layers. Although the Ag-Cu material system here does not fit a strict definition of a nanocomposite, the

presence of interface dislocations should not be overlooked. If dislocations that nucleated from the interface are driven back to their starting position it follows that inelastic recovery could occur as a result. Though not confirmed, the interface could act as a dislocation nucleation site during deformation and a dislocation storage location during unloading. The close proximity of the interfaces would allow for a greater number of dislocations to be driven back to the interface due to the small separation distance to the interface. Therefore, the high density of incoherent twin interfaces could play a dominant role in deformation, and the experimentally observed inelastic recovery.

Alternatively, the observed inelastic recovery could be driven by residual internal stresses due to inhomogeneous strains that develops due to the heterogeneous layer size (154 nm Cu vs 373 nm Ag). In ultra-fine-grained materials a strain mismatch occurs between low dislocation density regions such as the interior of a grain and the high dislocation density regions at the grain boundary [45]. It follows for the Ag-Cu layered material structure that the low dislocation density regions occur within the layer and those of high dislocation density at and near the incoherent twin interface. With the Ag phase having a nominal layer thickness twice that of Cu strain heterogeneity could take place due to the larger softer Ag phase deforming preferentially compared to the harder smaller Cu phase. Experimental work by Lonardelli et al. [25] investigating a bulk Al with a bi-modal grain size found that strain accommodated within smaller (nanocrystalline) grains was almost completely recovered due to internal stresses developed under loading. They also claim to show that depinning of dislocation need not be thermally assisted as has been proposed by molecular dynamics simulations of nanocrystalline materials [46] and in experimental studies of plastic recovery in nanocrystalline thin films [23,24]. Instead they propose if a sufficiently large strain forms that a portion of the plastic recovery, up to 28%

of a specimen loaded to 0.04 strain, can be recovered at room temperature. The proposed inelastic recovery, is promoted by high residual stresses which could provide the necessary driving force for dislocations to overcome energy barriers and be driven back to their nucleation sites.

A secondary source of internal stress which should be noted are coherency stresses at the cube-on-cube interface. Coherency stresses occur as a result of the lattice mismatch between Ag and Cu. While it could be possible for coherency stresses to drive dislocation motion during unloading their limited presence in the incoherent twin Ag/Cu interfaces would limit their role in promoting recovery.

As can be seen upon close inspection of Figure 4, there is a decrease in the strain-hardening, defined as $n = \partial(\ln \sigma) / \partial(\ln \varepsilon)$, with each deformation increment. The strain hardening exponent of the singularly loaded specimen is 0.10. For the incrementally loaded specimen, and specimens from the second casting, the strain hardening index of each increment is presented in Table 2. The transition of the strain hardening to a state of near-perfect plasticity (see loading increments 3 and 4 in Table 2) hints that the heterogeneous stress-state homogenizes with plastic deformation occurring throughout the specimen with increasing levels of accumulated strain. The transition in the stress-state could occur due to accumulation of dislocations at the interfaces. Dislocation transfer between layers is proposed to be increasingly difficult at elevated strains as the defect concentration at the interface increases and the Ag-Cu interface rotates away from the twin orientation relationship [20].

The experimental evidence points to recovery occurring under dynamic loading (10^3 s^{-1}) conditions but not under quasi-static loading (10^{-3} s^{-1}) conditions irrespective of the specimen thermal states explored here. The apparent rate dependence of the observed inelastic recovery

indicates that the responsible inelastic recovery mechanism(s) occurs at a rate greater than the quasi-static loading, but less than the dynamic loading. In this case, the mechanism(s) that are responsible for inelastic recovery may occur concurrently to the quasi-static deformation and would therefore not be observed after unloading. They also are likely to occur at length scales below those resolvable in the presented 3D-DIC measurements shown here.

Conclusions:

The inelastic recovery observed in the $\text{Ag}_{60}\text{Cu}_{40}$ cast eutectic material system is unique in that it is loading rate dependent and occurs over a time scale in which long term effects such as creep and diffusion cannot take place in sufficient quantities to account for the level of inelastic strain recovery observed. Additionally, the inelastic recovery mechanism does not have a temperature sensitivity in agreement with previous study of bi-modal structured Al. We have proposed a number of mechanisms and microstructural features that potentially play a collaborative role in the inelastic recovery observed based on the experimental evidence, namely, 1) the high density of incoherent twin interfaces and the associated stress field at the bi-phase boundary, 2) the heterogeneity of the stress-state within the material during deformation due to the size discrepancy of the Ag and Cu layers and the difficulty of communication of defects across the interface, and 3) loading rate, since recovery was only observed in dynamically deformed specimens. This study provides further support of inelastic recovery within FCC materials at the bulk-scale, adding to previous proposals that inelastic recovery could be a characteristic of appropriately structured FCC materials.

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Figure1

Figure 1: Microstructure of the 527 nm bilayer Ag-Cu material system (a) SEM micrograph showing the lamellar structure of eutectic colonies viewing parallel to the [101] growth direction, (b) layer thicknesses determined using a line intercept technique, averages indicated by bold blue (Ag) and red lines (Cu).

Figure2

Figure 2: Compressive stress-strain response of the 0°, 45°, and 90° loading orientations. The two curves from the 0° loading orientation are from separate castings. The markers used to identify each curve are also used along the bottom abscissa to indicate the dynamic strain ϵ_{Dy} (filled marker), and accumulated strain ϵ_A (open marker) for each curve.

Figure3

Figure 3: Dynamic stress-strain response of a 527 nm bilayer unidirectional Ag₆₀Cu₄₀ eutectic loaded parallel to the [101] growth direction. Filled markers on the bottom abscissa indicate the specimen dynamic strain. Open markers indicate the accumulated strain after each loading increment.

Figure4

Figure 4: Decreasing recovered inelastic strain with increasing levels of accumulated strain

Figure5

Figure 5: Incremental quasi-static loading and unloading of a unidirectional Ag₆₀Cu₄₀ cast eutectic.

Figure6

Figure 6: 3D DIC study of inelastic recovery under quasi-static loading. (a) A representative speckle patterned specimen. (b) The compliance corrected unloading curve (c,d,e,f) 3D-DIC calculated strains in the axial loading direction at the points indicated in (b). Point (f) occurs after a visible separation appears between the compression platens and specimen. The strain shows as positive because of the elastic recovery increases the overall length of the specimen.

Table1

Table 1: Deformation response of the 527 nm bilayer Ag-Cu loaded parallel to the [101] growth direction

Casting	Specimen	Loading Increment	Dynamic Strain (ϵ_{Dy})	Accum. Strain (ϵ_A)	Total Recov. Strain (ϵ_R)	Recov. Elastic strain (ϵ_E)	Recov. Inelastic strain (ϵ_I)	Δ Temp. ($^{\circ}$ C)
1	1	1	0.093	0.057	0.036	0.004	0.032	11.1
	2	1	0.106	0.075	0.031	0.004	0.027	11.8
		2	0.151	0.126	0.025	0.004	0.021	9.9
		3	0.170	0.160	0.010	0.004	0.006	6.2
		4	0.219	0.225	-0.009	0.004	-	8.6
2	1	1	0.068	0.050	0.018	0.004	0.014	6.7
	2	1	0.099	0.080	0.019	0.004	0.015	11.0

Table 2: Strain hardening response of 527 nm bilayer Ag-Cu loading along the [101] direction

Casting	Specimen	Loading Increment	Strain hardening index (n)
1	1	1	0.10
	2	1	0.16
		2	0.07
		3	0.01
		4	-0.02
2	1	1	0.13
	2	1	0.10

Directionally-cast Ag-Cu eutectic alloy
viewing along the [101] direction

Three loading orientations with respect to
the [101] crystallographic direction

Inelastic recovery along the 0° loading
orientation under dynamic conditions

Decreasing recovery with increasing strain