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# Precipitation Under Cyclic Strain in Solution-Treated Al-4wt%Cu I: Mechanical Behavior

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## Abstract

Solution-treated Al-4wt%Cu was strain-cycled at ambient temperature and above, and the precipitation and deformation behaviors investigated by TEM. Anomalously rapid growth of precipitates appears to have been facilitated by a vacancy super-saturation generated by cyclic strain and the presence of a continually refreshed dislocation density to provide heterogeneous nucleation sites. Texture effects as characterized by Orientation Imaging Microscopy appear to be responsible for latent hardening in specimens tested at room temperature, with increasing temperatures leading to a gradual hardening throughout life due to precipitation. Specimens exhibiting rapid precipitation hardening appear to show a greater effect of texture due to the increased stress required to cut precipitates in specimens machined from rolled plate at an angle corresponding to a lower average Schmid factor. The accelerated formation of grain boundary precipitates appears to be partially responsible for rapid inter-granular fatigue failure at elevated temperatures, producing fatigue striations and ductile dimples coexistent on the fracture surface.

## Introduction

Based upon past studies [1-3] significant changes in the precipitation behavior of Al-4wt%Cu seemed likely to occur under cyclic plasticity. Elevated point-defect concentrations due to deformation were expected to accelerate diffusion kinetics [2], and the presence of pinned dislocations to act as nucleation sites were expected to accelerate precipitate nucleation rates [4]. The role of glissile dislocations in precipitation under fatigue is less clear. Past studies have shown dissolution of Θ’ [5], and Θ” [6] precipitates under fatigue, and have postulated that precipitates may be softened by the introduction of anti-phase domain boundaries under deformation [7]. A transient increase in precipitate thickening rates has also been observed following monotonic deformation [8]. The combined effects of these phenomena upon cyclic-strain assisted precipitate growth from the solution state have never been investigated. Solution-treated material was desired for this study in order to attempt to understand the effects of cyclic strain. Material aged to contain fine precipitates has a tendency towards strain localization [7], leaving a great deal of uncertainty as to the strains experienced by any area inspected by TEM. Material aged to contain coarse precipitates would contain little remaining copper in the aluminum matrix, and thus a very low driving force for any precipitation during testing. Solution-treated material presented a clean slate with respect to precipitation, allowing for simple comparison of precipitation at different testing conditions, a high driving force for precipitation, and a tendency to strain more homogenously than material containing fine precipitates. Additionally, the role of texture hardening during precipitation hardening was investigated in an effort to deconvolute precipitation hardening from texture hardening.

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14. ABSTRACT
**Solution-treated Al-4wt%Cu was strain-cycled at ambient temperature and above, and the precipitation and deformation behaviors investigated by TEM. Anomalously rapid growth of precipitates appears to have been facilitated by a vacancy super-saturation generated by cyclic strain and the presence of a continually refreshed dislocation density to provide heterogeneous nucleation sites. Texture effects as characterized by Orientation Imaging Microscopy appear to be responsible for latent hardening in specimens tested at room temperature, with increasing temperatures leading to a gradual hardening throughout life due to precipitation. Specimens exhibiting rapid precipitation hardening appear to show a greater effect of texture due to the increased stress required to cut precipitates in specimens machined from rolled plate at an angle corresponding to a lower average Schmid factor. The accelerated formation of grain boundary precipitates appears to be partially responsible for rapid inter-granular fatigue failure at elevated temperatures, producing fatigue striations and ductile dimples coexistent on the fracture surface.**

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### Experimental Details

Two 12.7 kg (28 lb) ingots of 99.99% aluminum with 4 weight % copper added were cast by Alcoa, homogenized at 540ºC (1000ºF) for twelve hours, and hot rolled to 3/8" plate at an entry temperature of 400ºC (750ºF). Material was received in this condition and further cold rolled to 7 mm (0.275"), following which standard "dog-bone" specimens were machined at 0° and 45° from the rolling direction. Specimens were then dry ground by to a 600 grit finish and wet ground to a 1200 grit finish over a pane of glass. They were then annealed at 540ºC in molten salt for ten minutes and quenched into ice water immediately prior to mechanical testing. Orientation Imaging Microscopy (OIM) was performed on a FEI XL30 SEM equipped with TSL OIM 4.0 software. This showed a mixture of cube and fiber textures, with the cube texture predominant. Due to the very large areas sampled in order to gain statistical significance with a grain size of 350 µm, it was necessary to condense the data from multiple low-magnification scans to allow for an overall picture of the texture of the material. Pole figures are shown in Figure 1.

Following the generation of pole figures via the OIM software package, the raw data were further processed to generate a locus of the average Schmid factors (the purely geometric resolution of axial stress onto shear stress for a given slip system) of the material. This was achieved by transforming the Euler angles generated by the OIM system into Miller indices, and calculating the resolved stress on each slip system for any angle of application of unit stress in the plane of the plate between 0º from the rolling direction and 45º from the rolling direction, and then averaging the results for each angle across all the data points gathered.

Specimens machined with their stress axes parallel to the rolling direction of the parent plate were tested at plastic strain amplitudes of +/- 0.001 at 1 Hz, +/- 0.0025 at 0.4 Hz and +/-0.005 at 0.2 Hz and at temperatures of 25°C, 100°C, 175°C and 200°C in a forced hot air furnace on an Instron 1331 servo-hydraulic test system. The furnace was pre-heated and air flow started at the same time as the actuator, in order to ensure that the specimens began the test in a solution-treated condition.

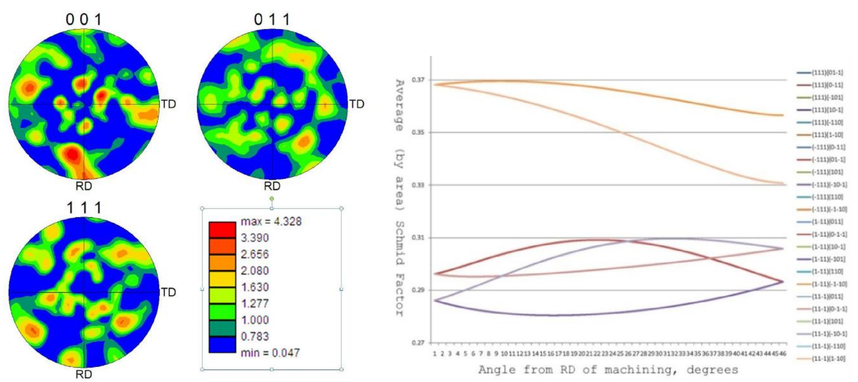


**Figure 1. Pole figures for the material as tested, and average Schmid factors for each slip system calculated from the OIM data. All slip systems have been plotted as a mathematical check despite the overlay between many systems.**

The accumulated plastic strain at failure for the specimen tested at $\varepsilon_p$=+/- 0.005 was used as a test-stop criterion for specimens tested at lower strain amplitudes. Thus each specimen accumulated equal plastic strain during the test, and each test duration was the same for a given temperature due to the inverse relationship between frequency and strain amplitude.

Specimens machined from their parent plate with their stress axes at 45° from the rolling direction were tested to failure at $\varepsilon_p$=+/- 0.001 at 1 Hz and $\varepsilon_p$=+/-0.0025 at 0.4 Hz.

## Results and Discussion

Cyclic Strain Behavior in the Rolling Direction

Cyclic hardening curves for specimens machined with their stress axes parallel to the rolling direction of the parent plate are presented in Figure 2. Of note in these figures is the softening and re-hardening of specimens tested at 175 °C and 200 °C. Due to the test procedure of starting the forced hot-air furnace at the same time as the actuator, all specimens tested at elevated temperature undergo a short ramp to temperature during the initial strain cycles of mechanical testing. The early softening is caused by recovery of cyclic hardening accumulated at lower temperatures, while the subsequent hardening is an effect of precipitation hardening occurring during testing. At 100 °C, although recovery is not apparent, precipitation hardening can be seen in the increasing stress through the plateau region as compared to tests run at 25 °C.

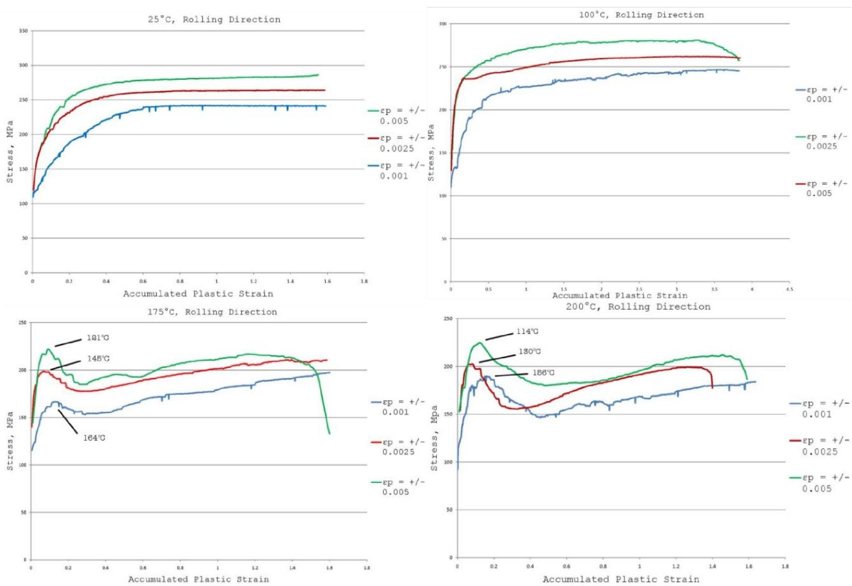


**Figure 2. Cyclic hardening curves for specimens tested parallel to the rolling direction of the parent plate. Note that the abscissa on each plot is accumulated plastic strain, and not cycles, to allow for comparison between different strain amplitudes.**

Cyclic Strain Behavior at 45° from the Rolling Direction

Cyclic hardening curves for specimens machined at 45° from the rolling direction and tested at $\varepsilon_p$=+/-0.001 at 1 Hz and $\varepsilon_p$=+/-0.0025 at 0.4 Hz are shown in Figure 2. Specimens oriented at 45° from the rolling direction show higher stresses for a given strain amplitude, and tend to fail at lower accumulated strains than specimens machined at 0° from the rolling direction. This effect is less pronounced at $\varepsilon_p$=+/-0.0025, 0.4 Hz than at $\varepsilon_p$=+/-0.001, 1 Hz. It appears that the difference between Schmid factors for secondary slip at 0° and 45° from the rolling direction roughly explains the difference in stresses for specimens strained at $\varepsilon_p$=+/-0.001, 1 Hz, while the difference in Schmid factors for primary slip aligns more closely with specimens strained at $\varepsilon_p$=+/-0.0025, 0.4 Hz. In light of this it seems likely, given the peak prior to the plateau stresses observed in tests performed at 25°C, that latent hardening is a significant factor in these observed texture effects, and is serving to block the activation of a second slip system at low strain amplitudes. The diminishing effect of orientation at $\varepsilon_p$=+/-0.0025, 0.4 Hz suggests that this may be the case. The effect of texture also becomes less pronounced at higher temperatures, suggesting that thermal activation of cross-slip or thermal climb may play a role in reducing the effects of texture under these test conditions. Additional confirmation for this hypothesis comes from the temperatures at which recovery becomes apparent in specimens tested at 175°C and 200°C. During the ramp to temperature early in specimen life, specimens machined from the parent plate at 45° to the rolling direction appear to recover at lower temperatures than specimens oriented in the rolling direction, suggesting a higher dislocation density for a given strain amplitude in specimens oriented at 45° from the parent plate. This effect also disappears at $\varepsilon_p$=+/-0.0025, 0.4 Hz. It appears that at high temperatures, where significant precipitation occurs, texture may change the effectiveness of precipitation hardening. At 200°C, it is apparent that texture hardening complements precipitation hardening, as specimens machined from the parent plate at 45° from the rolling direction appear to harden at a faster rate than those machined parallel to the rolling direction. This effect appears more subtly at 175°C, and is more apparent in the specimen strained at $\varepsilon_p$=+/-0.0025, 0.4 Hz. The likely explanation for this is that greater axial stresses are required for a sufficient force on a dislocation to cut a precipitate.

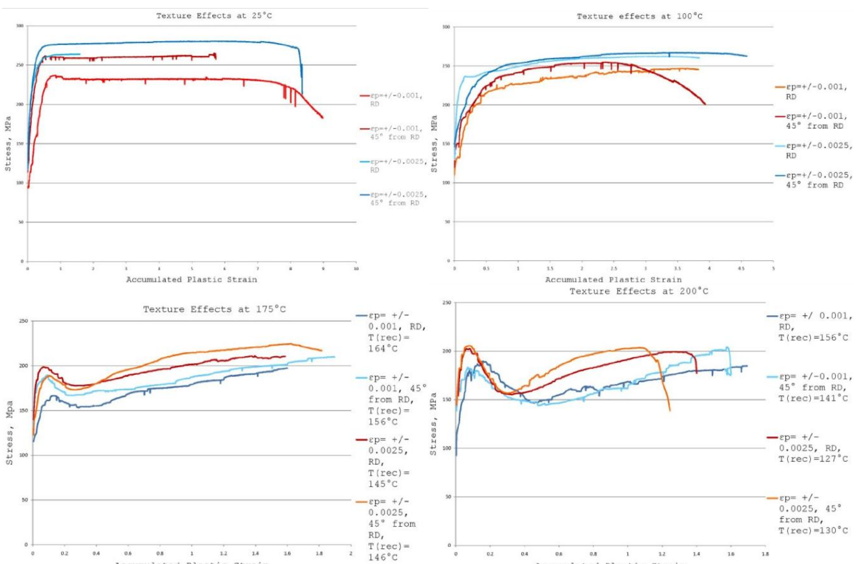


**Figure 3. Cyclic hardening curves for specimens machined at 45° from the rolling direction of the parent plate. Temperatures corresponding to the early peak are listed in the legend for specimens tested at 175°C and 200°C.**

Time-Dependent Effects at 200C

Several additional tests were performed at 200°C, in order to determine the effects of frequency and holds on the observed precipitation and mechanical behavior of the material. Frequency was reduced to 100 cycles/hour (0.027 Hz) for tests performed at $\varepsilon_p$=+/-0.005 and the test results compared against those generated at 0.2 Hz. Precipitation hardening is shown to dominate the hardening behavior of the material at 200°C via comparison of the results at 0.2 Hz and 0.027 Hz. By plotting results against time rather than accumulated plastic strain, and comparing the slopes of the cyclic hardening curves, the effect of strain hardening is shown to be minor relative to precipitation hardening. The strain accumulated by the 0.027 Hz test shown in Figure 4 would be insufficient to saturate the strain-hardening behavior at room temperature, and yet the slopes of the two curves are substantially similar. Another view of the same conclusion can be drawn from a 0.027Hz test incorporating strain holds. Also shown in Figure 3, the recovery behavior is dominated by precipitation hardening upon resumption of strain-cycling. That is: although the load drops during a hold, the cycle immediately following the hold reveals a stronger material, rather than a softened one.

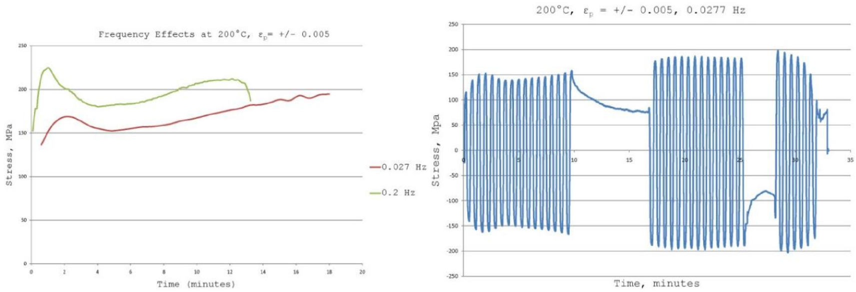


**Figure 4. Frequency effects and recovery behavior during holds at 200°C. Note the change in the abscissa to time from accumulated plastic strain to allow comparison of hardening effects. Samples sectioned from parent plate parallel to the rolling direction.**

Fracture Behavior.

At 25°C and 100°C, failure proceeded as expected, via nucleation and growth of a transgranular fatigue crack. At 175°C and 200°C, the increased diffusion of solute under cyclic strain promoted a ductile-intergranular fracture mode featuring both ductile dimples and fatigue striations on the fracture surface. It is believed that the presence of a narrow precipitate free zone accompanied by an accelerated growth of grain boundary precipitates is responsible for this fracture mode. Previous authors [3] have reported on the role of Θ' precipitation in the bulk in promoting grain boundary fracture, due to increased prevalence in multiple slip allowing for greater cooperative deformation across boundaries. The observed fracture mode persists at all strain amplitudes sampled at 175°C and 200°C, and persists in specimens of different textures. Initially, dynamic embrittlement was considered a possible cause of this fracture behavior, but referencing the strain-hold test shown in figure 3, it seems unlikely that hold crack plays a role, given the smooth stress drops and the absence of a faster stress drop in tension than compression. In specimens with a growing crack stopped prior to final failure, cooled to room temperature, and broken in monotonic tension, the same grain boundary failure mode persists, with ductile dimples apparent on the grain boundaries, and no striations observed in the region of monotonic failure. Examples of the fracture surface generated by this type of crack propagation can be seen in Figure 4.

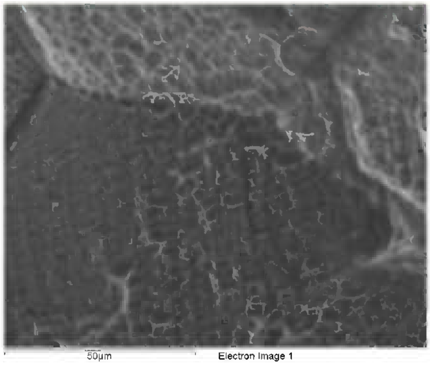


**Figure 5. Scanning electron micrograph, showing ductile dimples and fatigue striations on the fracture surface of a specimen machined at 45° from the parent plate and tested to failure (800 cycles) at $\varepsilon_p$=+/-0.001, 1 Hz, 200°C.**

## Conclusions

1. Precipitation hardening dominates the high-temperature cyclic strain behavior of Al-4wt%Cu from the solution state, due to accelerated formation of Θ' precipitates.
2. Enhanced precipitation kinetics described in a companion paper promote the formation of ductile dimples in the precipitate free zone, and precipitation hardens the surrounding material, enhancing strain localization at grain boundaries at 175°C and 200°C leading to intergranular failure by a hybrid stage II fatigue crack and void coalescence mechanism.
3. Texture hardening appears to diminish with both increasing temperature and increasing strain amplitude, but is accentuated by precipitation.

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