## Title

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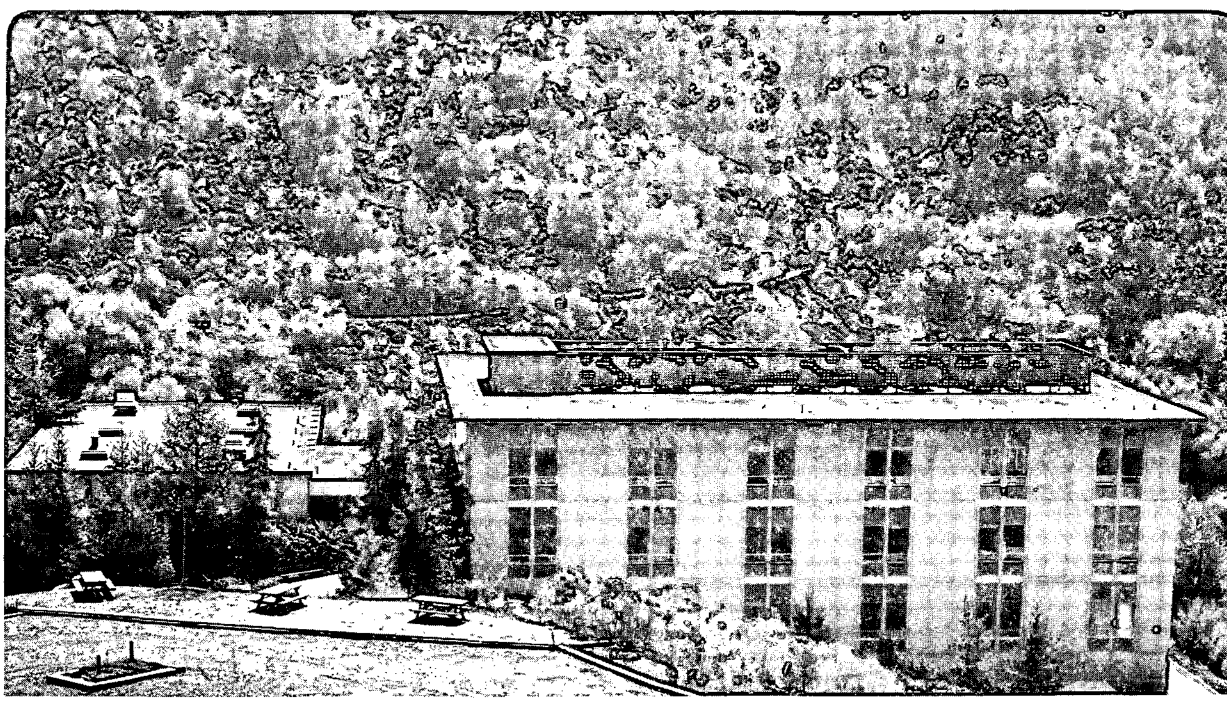



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# Influence of Grain Structure and Solute Composition on the Work Hardening Behavior of Aluminum at Cryogenic Temperatures

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# Influence of Grain Structure and Solute Composition on the Work Hardening Behavior of Aluminum at Cryogenic Temperatures

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An unrecrystallized structure is found to significantly improve the work hardening characteristics by lowering the work hardening rate during the early stages of deformation. This is in contrast to a recrystallized structure, which requires a higher work hardening rate to accommodate the greater degree of multiple slip necessary to maintain strain compatibility between the more randomly oriented grains. The stronger texture associated with the unrecrystallized structure allows deformation to occur more efficiently. The addition of magnesium also improves the work hardening characteristics by increasing the overall level of the work hardening rate. The improved characteristics of the work hardening behavior result in a parallel increase in both the strength and ductility at cryogenic temperatures. These findings are positive since they suggest a method by which improvements in the work hardening behavior and subsequent mechanical properties may be obtained through practical modifications of the microstructure and composition.

## INTRODUCTION

The initial selection of a material for any structural application at cryogenic temperatures is most often governed by its combination of strength and fracture toughness. Since the strength and fracture toughness almost invariably exhibit an inverse relationship with each other, it becomes necessary to optimize this combination to provide maximum strength and toughness values. Unfortunately, our current understandings of the factors that control the strength and toughness are limited, thus making the successful optimization process a fortuitous occurrence rather than one resulting from "good science".

Continuing research on the mechanisms controlling yield and fracture[1-5] show that the first concern in interpreting the temperature dependence of the strength-toughness characteristic is the possibility of a change in the fracture mode. The most familiar fracture mode change is the ductile-brittle transition, where fracture usually occurs by a microvoid coalescence mechanism at high temperatures but changes to either transgranular cleavage or intergranular separation at temperatures below the ductile-brittle transition temperature. Upon the preservation of a ductile fracture mode, the mechanisms controlling yield and fracture are directly associated with the work hardening behavior[1,2,5]. This is most readily seen by considering the various ductile fracture theories which all lead to models of the general form

$$K_{Ic} \sim \varepsilon_f \sqrt{E\sigma_y}, \qquad (1)$$

where E is the elastic modulus, $\sigma_y$ is the yield strength, and $\varepsilon_f$ is the strain to failure. Although these models suggest that the fracture toughness and yield strength should vary together, the strain to failure in many aluminum alloys is strongly dependent on the work hardening behavior.

The relationship between strength, elongation, and work hardening can be observed through overlaid plots of the work hardening rate, $d\sigma/d\varepsilon$, and the true stress, $\sigma$, versus the true strain, $\varepsilon$. Such plots have been used extensively in previous research efforts to study the macroscopic tensile behavior of aluminum-lithium alloys at cryogenic temperatures[1,2,5-7]. Geometric instability or "necking" in tension occurs when the strain hardening rate equals the true stress ($d\sigma/d\varepsilon = \sigma$)[8]. For the alloys studied by Glazer, et al.[1,2], and Chu, et al.[5-7], this instability is found to coincide with failure. From the schematic in Figure 1a, it is apparent that the coupling between the work hardening rate and the flow stress determines the strain to failure. This relationship is particularly true when considering a change in test temperature. In general, an increase in the yield strength occurs with decreasing temperature, suggesting an increase in fracture toughness by Equation (1). However, an increase in the yield strength with no change in the work hardening behavior would lead to a decrease in the strain to failure that may be significant enough to decrease the fracture toughness rather than improve it.

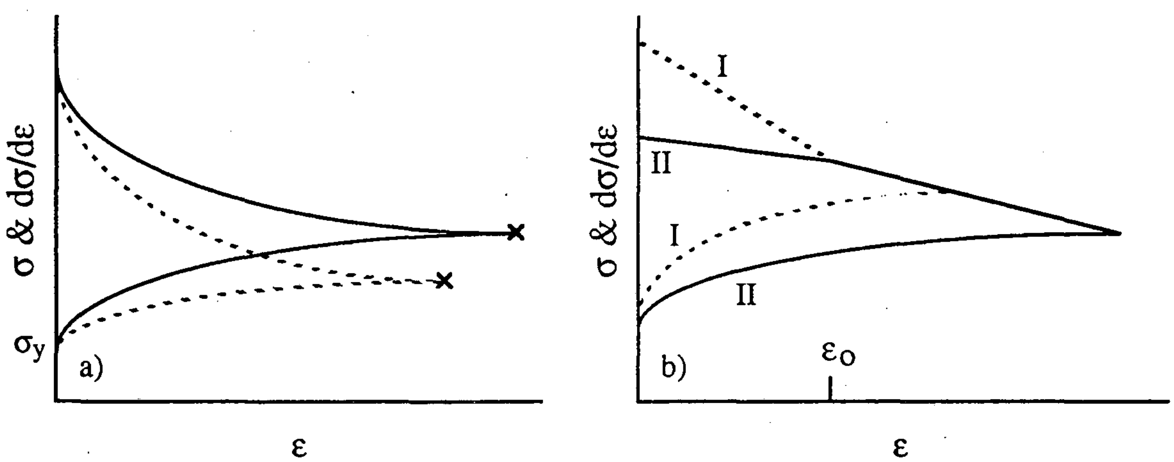


**Figure 1.** Schematic illustrating the influence of the work hardening behavior on the elongation. An increase in the overall level of the work hardening rate is shown in figure a) whereas a lowering of the early work hardening rate is illustrated in figure b).

From the discussion above, it is apparent that the work hardening behavior plays an important role in determining the mechanical properties of a material. Subsequent efforts in the area of work hardening have been made in hopes of producing a model that can accurately predict the mechanical behavior provided a set of parameters that describe the microstructure of any given material[9-11]. One widely accepted model presented by Mecking, Kocks, and coworkers[10,12] suggests that the dominating microstructural feature is the dislocation density. This model requires the material to be in a homogeneous state; in particular, the arrangement of dislocations must be homogeneous which for most materials, necessitates extensive deformation (greater than 5 to 10% strain).

For structural materials, however, the work hardening behavior prior to the regime described by Mecking's model may be of greater importance. To illustrate this, consider the schematic comparison in Figure 1b. Both Alloys I and II exhibit the same yield strength, but different work hardening characteristics during the early stages of deformation. Beyond $\varepsilon_o$, the work hardening behavior is assumed to be similar. Because of the higher work hardening rate in the early stages, the flow stress in Alloy I increases more rapidly, thus causing the material to reach the necking criterion much earlier than Alloy II. Equation (1) suggests that an increase in the fracture toughness would result in the Alloy II.

A recent work hardening study on the aluminum-lithium alloy 2090 suggests that this early stage of work hardening may be largely influenced by the grain structure, namely the grain size, grain shape, and texture[7]. Intuitively the grain structure should influence the early development of the dislocation array preceding a homogeneous arrangement of dislocations. It is also conceivable that other microstructural parameters such as the solute composition, precipitate type, and precipitate volume fraction may play an important role during this early development. If such a relation between these microstructural features and the early work hardening behavior does exist, it may provide a reliable means by which the work hardening behavior and subsequent strength-toughness combination can be altered through controlled modifications of the microstructure.

The work presented in this paper is part of a continuing effort toward understanding the manner by which these various microstructural features influence the subsequent work hardening characteristics. In particular, an investigation on the influence of grain structure and magnesium in aluminum is undertaken.

## EXPERIMENTAL PROCEDURE

The materials used in this study consisted of pure aluminum and aluminum containing 2.5 weight percent magnesium. Both materials were cold rolled 50% to a 3mm thickness after sufficient hot rolling at appropriate temperatures. Flat tensile specimens were machined from the sheets and then heat treated to produce a recrystallized grain structure. Heat treatment times and temperatures were selected to produce grain sizes of 125 and 250 μm. A heat treatment was also selected for pure aluminum samples to produce a partially recrystallized grain structure consisting of approximately 50% equiaxed grains and 50% unrecrystallized regions. All sides of the tensile gage were polished to a 14 μm finish prior to testing. Selected samples were polished to 0.05 μm to facilitate the observation of slip relief patterns on the surface through optical microscopy.

All tensile tests were conducted in displacement control. Displacement rates corresponded to strain rates of approximately $10^{-4}$ per second within the plastic range. Engineering stress and strain data were collected via computer and then converted to true stress and strain values. The instantaneous strain hardening rate, $d\sigma/d\varepsilon$, was determined by means of a point to point differentiation on a cubic spline fit representing the true stress true strain curve.

Strain hardening curves are presented as a function of plastic stress ($\sigma - \sigma_y$) in order to eliminate differences in yield strength. Such plots have been utilized to separate and identify the effect of temperature[1,2,5], and to some degree the microstructure[5-7], on the development of work hardening for tensile specimens. Plots of this form typically exhibit a decreasing linear relationship between the work hardening rate and the flow stress, which Mecking, Kocks, and coworkers attribute to the competition between an athermal dislocation storage term and a temperature-sensitive, dynamic recovery term[9,10,12].

## RESULTS AND DISCUSSION

**Table 1.** Yield strength, ultimate strength, and uniform elongation obtained for the materials tested.

| Grain Size (μm) | | partially recrystall. | | 125 | | 250 | |
|---|---|---|---|---|---|---|---|
| Temperature (K) | | 300 | 77 | 300 | 77 | 300 | 77 |
| Al | $\sigma_y$ (MPa) | 53.5 | 66.5 | 11.0 | 14.5 | 9.5 | 12.0 |
| | $\sigma_u$ (MPa) | 71.5 | 171.5 | 49.5 | 138.0 | 44.5 | 128.5 |
| | $\varepsilon_u$ (%) | 20.9 | 34.0 | 32.9 | 32.7 | 32.5 | 32.0 |
| Al-2.5%Mg | $\sigma_y$ (MPa) | | | 56.5 | 62.1 | 48.0 | 60.0 |
| | $\sigma_u$ (MPa) | | | 178.0 | 279.0 | 158.5 | 259.0 |
| | $\varepsilon_u$ (%) | | | 21.1 | 35.3 | 22.4 | 36.2 |
| Al-5.0%Mg | $\sigma_y$ (MPa) | | | 92.0 | 91.5 | 81.5 | 87.0 |
| | $\sigma_u$ (MPa) | | | 250.5 | 363.0 | 229.0 | 332.5 |
| | $\varepsilon_u$ (%) | | | 23.1 | 38.3 | 39.9 | 44.9 |

## *Influence of Grain Structure*

Table 1 lists the mechanical properties obtained for the materials tested. Note the elongation values obtained for the fully recrystallized pure aluminum samples; both show a slight decrease with decreasing temperature. In contrast, the elongation value determined for the partially recrystallized pure aluminum specimen exhibits a dramatic increase with decreasing temperature. Since no compositional influences exist, this difference in temperature dependence must be attributed to the difference in grain structures. The contrast between the two grain structures can also be observed through comparison plots of $d\sigma/d\varepsilon$ versus $\sigma - \sigma_y$ (Figure 2); the partially recrystallized structure exhibiting a much lower work hardening rate during the early stages of deformation.

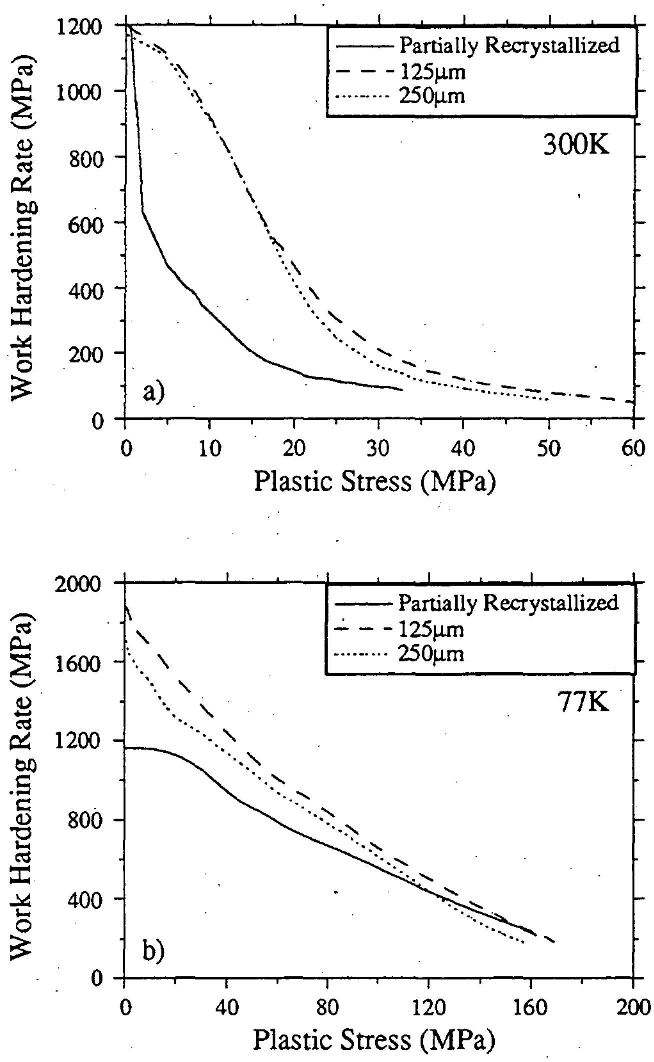


**Figure 2.** Plots of $d\sigma/d\varepsilon$ versus $\sigma$-$\sigma_y$ for pure aluminum with partially recrystallized grain structure and fully recrystallized grain structure with labeled grain sizes at a) 300K and b) 77K.

A comparison of Figure 2 with the schematic in Figure 1b suggests that the partially recrystallized structure should exhibit a greater elongation; however, at 300K, the large difference in yield strengths (Table 1) between the two structures prevents this from occurring. At 77K, the difference in yield strengths is sufficiently small to allow the partially recrystallized structure to exhibit a greater elongation than the fully recrystallized structure. A comparison plot of the strain hardening rate versus true strain for the two grain structures (Figure 3) shows that the rapid drop to lower work hardening rates is associated with a greater degree of perfectly-plastic behavior.

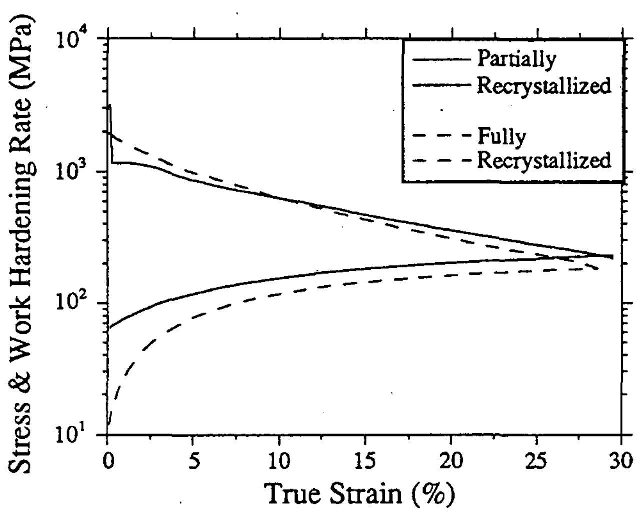


**Figure 3.** Composite plots of dσ/dε and σ versus ε obtained from pure aluminum samples illustrating the influence of partially recrystallized and fully recrystallized grain structure (125μm grain size) on the stress-strain properties at 77K.

Slip relief patterns taken from the partially recrystallized specimen at consecutive strains (Figure 4) suggest that the controlling deformation mechanism resides within the grain structure rather than the dislocation structure. The greater roughness associated with the recrystallized region upon extensive deformation is due to the difference in initial texture. In order to maintain strain compatibility between the more randomly oriented grains, the recrystallized structure must exhibit a greater degree of multiple slip[13], particularly during the early stages of deformation, which produces a rougher slip profile. Previous studies on the aluminum-lithium alloy 2090 have shown that such extensive slip relief at the granular level is associated with a higher work hardening rate[14].

Further analysis of Figure 2 shows that the work hardening curves can be separated into two approximately linear regimes which suggests the existence of two differing mechanisms. This is most evident at 300K. From the results discussed above, the earlier regime appears to be associated a greater degree of multiple slip. Although less distinct, this earlier mechanism also exists at 77K. It is important to note that the latter regime appears to fall roughly on the same line for all three materials, suggesting that the difference in grain structure is limited in its ability to influence the work hardening behavior to only the early stages.

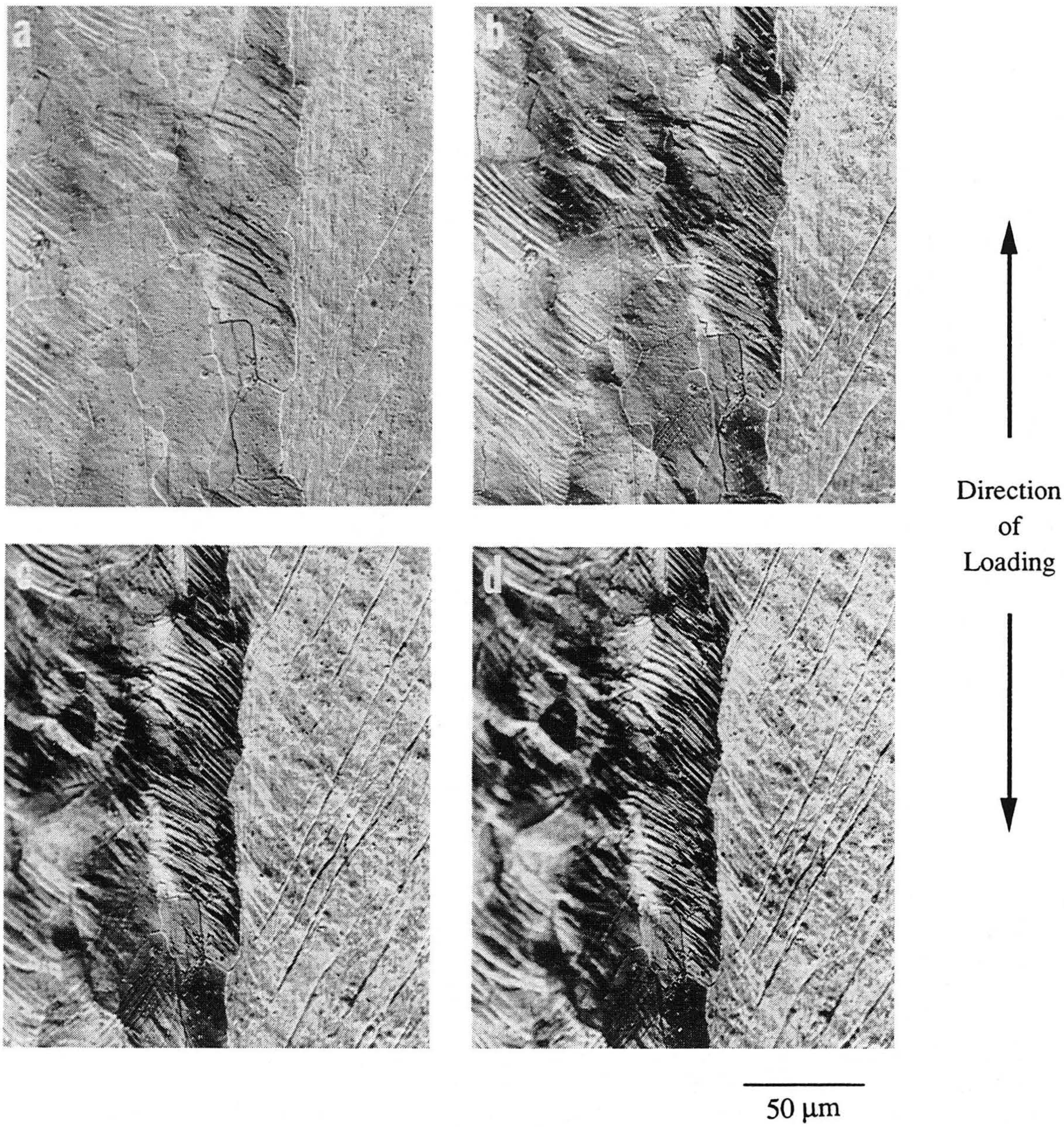


**Figure 4.** Optical micrograph showing the evolution of slip relief at 300K in both the unrecrystallized (right) and recrystallized (left) regions after a) 2%, b) 5%, c) 10%, and d)15% plastic strain. Note the greater roughness exhibited by the recrystallized structure. (XBB 936-3999)

The combined results of Figures 2 through 4 indicate that a higher work hardening rate is necessary to deform recrystallized grains due to the greater degree of multiple slip required to maintain strain compatibility. It follows that an unrecrystallized structure is more efficient in accommodating deformation; a smaller increase in stress is required relative to a recrystallized structure to produce a given amount of strain. There is evidence that such differences in grain structure may also influence the cryogenic fracture toughness. Studies on the cryogenic fracture properties of aluminum-lithium alloy 2090 reveal a drop in the fracture toughness with the introduction of a recrystallized structure[2,5].

*Influence of Magnesium*

A second look at Table 1 shows that in all cases, the addition of magnesium and testing at cryogenic temperatures are accompanied by an increase in both yield and ultimate strengths. This increase is typical of solution hardening. The elongation, however, does not exhibit the same trend. Specimens tested at 300K exhibit a 30% drop in the elongation with the addition of magnesium. In contrast, specimens tested at 77K exhibit an increase in the elongation with magnesium content. Jata and Starke suggest that at cryogenic temperatures, the magnesium addition produces greater slip homogeneity[15], which results in an increase in ductility[16].

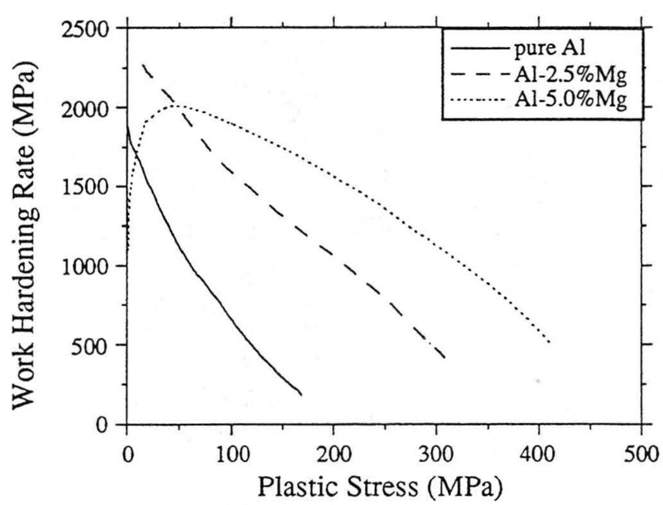


**Figure 5.** Variation of work hardening rate with magnesium content at 77K. (Initial drop in work hardening rate for the Al-5.0%Mg sample is due to dislocation trapping by magnesium solutes resulting in an initial interval of easy glide. Amount of strain attributed to this easy glide is estimated to be about 0.1%.)

Increases in the work hardening rate due to magnesium at cryogenic temperatures have been observed in earlier research efforts but have usually been associated with increases in the ultimate strength rather than increases in the elongation[17]. Figure 5 shows that the improved elongation at cryogenic temperatures can be attributed to an overall increase in the work hardening rate during the entire deformation range. However, this is not necessarily sufficient. The results of Figures 2 through 4 indicate that a greater work hardening rate, particularly during the early stages of deformation, should result in a decrease in the elongation. However, unlike in pure aluminum, where the latter linear regime falls at roughly the same level for both microstructures, Figure 5 shows that the addition of magnesium decreases the rate at which the work hardening curve decreases with flow stress. In other words, the addition of magnesium increases the ability of the alloy to maintain higher work hardening rates throughout the deformation process.

## CONCLUSION

The majority of research efforts on work hardening have ignored the difference between an increased work hardening rate and an improved work hardening behavior. Parallel increases in strength and ductility at cryogenic temperatures are often associated with increases in the overall level of the work hardening rate. Such improvements are observed to occur with the addition of magnesium. However, this is only one means of improving the work hardening behavior. A second method, which may provide the key to high strength-toughness combinations at cryogenic temperatures resides in lowering the work hardening rate during the early stages of deformation. The grain structure plays an important role in this case: an unrecrystallized structure exhibiting a lower early work hardening rate relative to a recrystallized structure. Experimental evidence shows that the recrystallized structure exhibits a higher work hardening rate due to the greater degree of multiple slip necessary to maintain strain compatibility between the more randomly oriented grains. The stronger texture associated with a partially recrystallized structure reduces the degree of multiple slip, thereby lowering the work hardening rate during the early stages of deformation.

The results obtained from this study are promising, as they suggest practical means by which improvements in the work hardening behavior and subsequent mechanical properties can be obtained through modifications of the microstructure and composition.

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