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# Elevated temperature deformation behavior of nanostructured Al–Ni–Gd–Fe alloys

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## Abstract

The elevated temperature deformation behavior of nanostructured Al<sub>89</sub>Ni<sub>3</sub>Gd<sub>7</sub>Fe<sub>1</sub> alloy was characterized. Tensile strength was 760 MPa at 373 K. Ductility of the alloy increases with increasing strain rate at 573 K. At high temperatures (623–673 K), the operative deformation mechanism is dislocation-climb controlled.

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*Keywords:* Aluminum alloys; High temperature deformation; Ultrafine grained microstructure; Nanostructured alloys

## 1. Introduction

The promise of non-equilibrium processing has resulted in a major thrust for the development of high performance Al-based alloys with enhanced strength, stiffness and thermal stability at elevated temperatures. For high temperature applications, the focus is on producing alloys with a high volume fraction of dispersoids and very fine microstructures [1,2]. Contributions to strength enhancement of the soft metal matrix arise because of non-shearable barriers to the movement of dislocations. In particular, crystallization of amorphous alloys provides opportunities to produce bulk nanostructured materials with a high volume fraction of intermetallic particles. Phase transformation from the amorphous state has some unique features [3]: (a) homogenous nucleation, (b) high nucleation frequency, (c) low growth rate, and (d) nanoscale interparticle spacing. All of these are beneficial for developing high-

strength dispersion-strengthened alloys. It also enables great flexibility in the design of materials with chosen volume fractions, types, and sizes of dispersoids and of alloying additions. Amorphous aluminum alloys can be obtained in Al–TM–RE (TM = transition metals, RE = rare earth elements) systems [4,5]. Shiflet and coworkers [6] have reported that Al–Ni–Fe–Gd alloys have unique metallic glass formability. So far, ambient temperature mechanical properties of such alloys have been reported and very little work has been done on determining the elevated temperature tensile behavior of bulk nanocrystalline alloys [7,8].

The purpose of this paper is to report the elevated temperature deformation behavior of an as-extruded dispersion-strengthened Al<sub>89</sub>Ni<sub>3</sub>Gd<sub>7</sub>Fe<sub>1</sub> (at.%) alloy at various temperatures and strain rates.

## 2. Experimental procedure

Atomized Al<sub>89</sub>Ni<sub>3</sub>Gd<sub>7</sub>Fe<sub>1</sub> powders were extruded into 15.9 mm diameter rods. The starting billet temperature was in the range from 733 to 761 K, with 10 min

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of heat-up time and 4 min soaking time. An extrusion ratio of 20:1 was used for all rods. Mini-tensile specimens with rectangular cross-section (gage length 1.3 mm and gage width 1.0 mm) were electro-discharge machined from as-extruded rods in the longitudinal direction. The tensile specimens were polished to the final thickness of  $\sim 0.5$  mm and 1  $\mu\text{m}$  finish. Tension tests were performed on a custom-built, computer-controlled mini-tensile tester in the temperature range of 373–673 K and initial strain rate range of  $5 \times 10^{-5}$  to  $1 \times 10^{-2} \text{ s}^{-1}$ .

Microstructure of the extruded rod was examined using a Philips EM430 transmission electron microscope (TEM) at 300 kV. Thin foils were prepared from as received alloys by double jet electropolishing using a Struers-Tenupol 2 device operating at  $\sim 12$  V. The electrolyte was 20 vol%  $\text{HNO}_3$  and 80 vol% methanol cooled to 243 K. The effect of temperature on the tensile behavior of this alloy was analyzed based on the fractographic examinations, which were conducted using a Hitachi S4700 scanning electron microscope (SEM).

### 3. Results and discussion

#### 3.1. Microstructure

Fig. 1 shows the microstructure of an as-extruded  $\text{Al}_{89}\text{Ni}_3\text{Gd}_7\text{Fe}_1$  alloy. Hot extrusion of the amorphous powders results in the formation of ultra-fine structure consisting of  $\sim 35$  vol% of nanostructured intermetallic particles, embedded in the matrix. The matrix was almost pure  $\alpha$ -Al due to very low solid solubility of Gd, Ni, and Fe in Al. The average grain size was

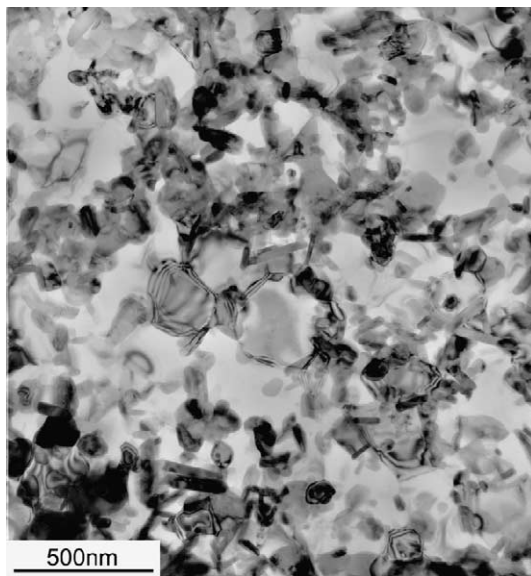


Fig. 1. Bright-field TEM micrograph of as-extruded  $\text{Al}_{89}\text{Ni}_3\text{Gd}_7\text{Fe}_1$  alloy.

$\sim 200$  nm. Rod-like particles (length  $\sim 160$  nm and width  $\sim 30$  nm) and equiaxed particles ( $\sim 80$  nm) were mostly located along the grain boundaries. Dislocation density was very low in the interior of the grains.

#### 3.2. Elevated temperature deformation behavior

The variation of tensile behavior with temperature at an initial of strain rate  $1 \times 10^{-3} \text{ s}^{-1}$  is shown in Fig. 2, where UTS is the ultimate tensile strength, YS the yield strength, and  $\epsilon_f$  the failure strain (true plastic strain from the stress–strain curves). It can be noted that the tensile strength of this alloy is 760 MPa at 373 K, which is higher than that of traditional elevated temperature alloys. For example, the tensile strength of extruded 2618 aluminum alloy is 405 MPa at 373 K [9]. Strength of  $\text{Al}_{89}\text{Ni}_3\text{Gd}_7\text{Fe}_1$  alloy decreases gradually with increasing temperature and the ductility increases sharply above 623 K and reaches 24% elongation at 673 K (at room temperature the material failed in a brittle manner). Retention of strengthening at elevated temperatures can be attributed to the high volume fraction of nanostructured dispersoids. Also the nanostructure reduces the size of nucleating flaws and increases the resistance to crack propagation, leading to higher fracture strength [10]. Very fine second phase particles situated at the grain boundary in this alloy can be effective in the prevention of grain boundary migration, limiting grain growth due to the Zener pinning. Because of the few intragranular particles, Orowan dislocation looping cannot be considered as the primary strengthening mechanism. However, for such nanostructured materials, the theoretical framework for dislocation–particle interaction has not been developed. For example, the strengthening due to the interaction of dislocations with grain boundary particles has not been quantified.

True stress–strain curves for various initial strain rates at 573 K are shown in Fig. 3. At this temperature, the yield stress and failure strain increases with increasing strain rate. Similar behavior was also observed in

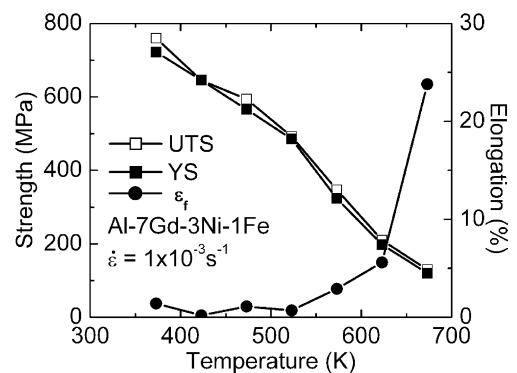


Fig. 2. Variation of strength and ductility with temperature for  $\text{Al}_{89}\text{Ni}_3\text{Gd}_7\text{Fe}_1$  alloy.

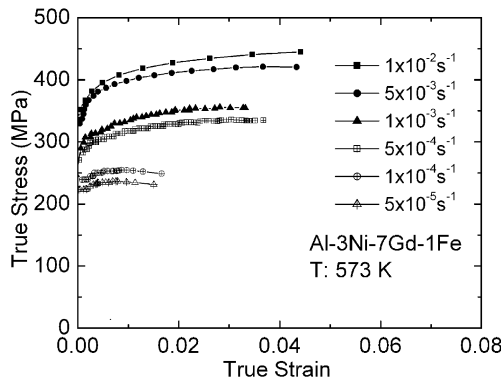


Fig. 3. Stress–strain behavior of Al<sub>89</sub>Ni<sub>3</sub>Gd<sub>7</sub>Fe<sub>1</sub> alloy at 573 K for various initial strain rates.

rapid solidification/powder metallurgy (RS/PM) aluminum alloys [11]. This is quite different from that of ingot metallurgy processed aluminum alloys with coarser microstructures. It has previously been shown that in materials with particles present along grain boundaries, plastic flow is produced by dislocation nucleation from the grain boundaries and from constituent particles pinning the grain boundaries [12]. The interaction between dislocations and particles results in more damage of the grain boundaries at lower strain rates. For Al<sub>89</sub>Ni<sub>3</sub>Gd<sub>7</sub>Fe<sub>1</sub> alloy, uniform elongation decreases from 4% to 1% when initial strain rate decreases from  $1 \times 10^{-2} \text{ s}^{-1}$  to  $5 \times 10^{-5} \text{ s}^{-1}$  at 573 K, which implies an early start of the failure mechanisms with decreasing strain rate. Cavity nucleation and strain localization due to insufficient work hardening are possible explanations for the observed behavior. At lower strain rates, flow stress increases initially and then decreases with increasing strain. It is possible that the rate of dynamic recovery or evolution of damage leads to flow softening.

The variation of flow stress with strain rate at high temperatures is shown in Fig. 4. The apparent stress exponent decreases with increasing temperatures. The observed stress exponents are higher than 5. The presence of particles at the grain boundaries makes elevated

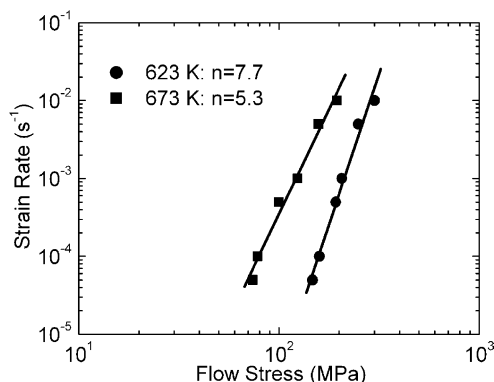


Fig. 4. Variation of strain rate with flow stress at high temperatures.

temperature deformation more difficult. However the values are not like other dispersion-strengthened Al-based alloys, in which very high apparent stress exponents were observed [1]. A possible explanation is that most of particles are present along the grain boundaries. In this case, it is assumed that the strain rate is controlled by the breakaway of grain boundary dislocations from the particles, and the stress exponent could be reduced by  $\sim 1/4$ – $1/9$  compared to the microstructure containing particles within the grains [13].

The deformation behavior of dispersion-strengthened materials at high temperatures can be represented by the constitutive equation which incorporates threshold stress as given by [14,15]

$$\dot{\epsilon} = A \left( \frac{GbD_L}{kT} \right) \left( \frac{\sigma - \sigma_0}{G} \right)^n \quad (1)$$

where  $\dot{\epsilon}$  is the strain rate,  $D_L$  the lattice self-diffusivity,  $G$  the shear modulus,  $b$  the Burgers vector,  $k$  the Boltzmann’s constant,  $T$  the absolute temperature,  $\sigma$  the applied stress,  $\sigma_0$  the threshold stress,  $A$  the dimensionless constant,  $n$  the true stress exponent. The threshold stress concept is often introduced to rationalize the high stress exponents in dispersion-strengthened alloys [16]. Lagneborg and Bergman [17] introduced the widely used method to determine experimental threshold stress by plotting  $\dot{\epsilon}^{1/n}$  versus  $\sigma$  with  $n = 5$  for pure aluminum, and extrapolating the linear fitted line to zero  $\dot{\epsilon}$ . Experimental threshold stress values at high temperatures are listed in Table 1. It can be noted that it decreases with temperature. In the climb threshold models, the origin of the threshold stress is considered as the increase in dislocation line length during the climb bypass [18–21]. The threshold stress models proposed by Arzt and coworkers [22,23] and Mishra et al. [24] suggest that the origin of threshold stress is due to the attractive dislocation–particle interaction. These models for threshold stress for dislocation creep are based on interaction of lattice dislocations with particles within the grain. However, as noted earlier, in the present alloy particles are mostly on the grain boundaries. Existing threshold models cannot predict threshold stress for this kind of nanostructured alloy.

The normalized strain rate,  $\dot{\epsilon}kT/D_LGb$ , is plotted against the normalized effective stress  $(\sigma - \sigma_0)/G$ , on double logarithmic scales in Fig. 5. For the data analysis, the values of the lattice self-diffusivity  $D_L [\text{M}^2/\text{s}] = 1.71 \times 10^{-4} \exp(-142/RT)$  [25], and  $G [\text{MPa}] = 3.0 \times 10^4 - \text{ce:hsp sp}="0.25"/>16T$  [26], where  $R$  is the universal gas constant, were used. After considering

Table 1  
Experimental threshold stress values at high temperatures

| Temperature (K)        | 623 | 673 |
|------------------------|-----|-----|
| Threshold stress (MPa) | 69  | 6   |

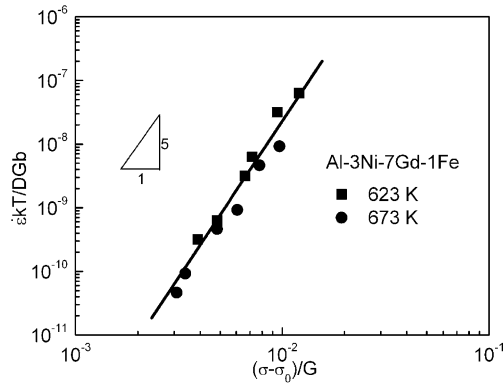


Fig. 5. Temperature and diffusivity compensated strain rate versus normalized effective stress.

threshold stress, the experimental results at 623 and 673 K can be fitted well into the straight line with slope 5, which indicates the dislocation-climb controlled deformation. The value of dimensionless constant  $A$  in Eq. (1) is 243.

SEM fractographs of tested samples at 573, 623 and 673 K and initial strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$  are shown

in Fig. 6. The present results show transition in fracture surface morphology from transgranular to intergranular in the temperature range 573–673 K. At 573 K, shallow ductile dimples with different sizes are present (Fig. 6(a) and (b)). It can be noted that the dimple size is close to the grain size. In very small dimples, dispersoids responsible for void initiation can be observed. At 623 K, the dimples are deeper and larger than those at 573 K (Fig. 6(c) and (d)). A number of particles are visible within the dimples and along the edge of the dimples. The characteristic dimple size is around  $1.2 \mu\text{m}$ . At 673 K, dimple size ranges from 1 to  $4 \mu\text{m}$  (Fig. 6(e) and (f)). There are some less well-developed dimples with a discontinuous dimple perimeter. However, individual grains can be identified at the bottom of the dimples at higher magnification as shown in Fig. 7. It appears that intergranular fracture starts contributing at this temperature. This fracture mode has been observed in aluminum alloys at low stresses and high temperatures. Growth of grain boundary voids by coupled diffusion and power-law creep leads to intergranular fracture. It covers a large part of the fracture maps proposed by Ashby et al. [27].

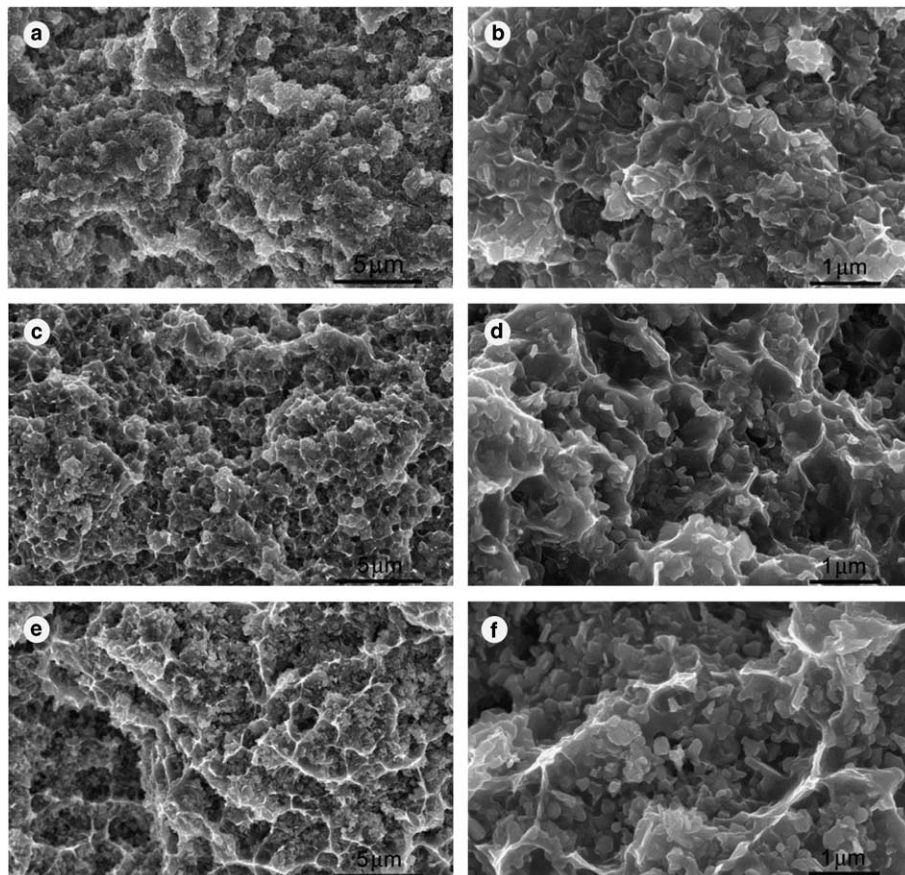


Fig. 6. SEM fracture surface of tested at: (a, b) 573 K; (c, d) 623 K; and (e, f) 673 K.

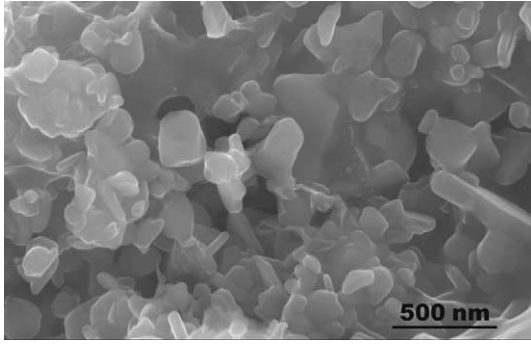


Fig. 7. High magnification SEM fracture surface of deformed sample at 673 K.

#### 4. Conclusions

1. A high volume fraction of nanostructured intermetallic dispersoids can be obtained by the use of the amorphous precursor, which enables the  $\text{Al}_{89}\text{Ni}_3\text{Gd}_7\text{Fe}_1$  alloy to exhibit high elevated temperature strength.
2. At 573 K, the ductility increases with the increasing strain rate.
3. The apparent stress exponent was higher than 5, indicating dislocation-climb mechanism with temperature-dependent threshold stress.
4. The fracture surface appearance varies with temperature. The fracture features change from transgranular to intergranular in the temperature range 573–673 K.

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