Elevated-Temperature Coarsening Behavior in Aluminum Alloys

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Coarsening kinetics of the equilibrium phase in the Al-rich end of the Al-Ti-Cu and Al-Ti-Ni systems have been studied using transmission electron microscopy as a function of annealing times at 698 K. The equilibrium phase in the Al-Ti-Ni system coarsens at a rate that is 6 times faster than that in the Al-Ti-Ni phase. This difference in the coarsening kinetics is related to the amount of mismatch of each of the equilibrium phases with respect to the Al-matrix.

Keywords
aging, aluminum alloys, electron microscopy, structural analysis

1. Introduction

RAPID QUENCHING of molten metals and alloys has been shown to result in the formation of several microstructures. Some of these microstructures contain nonequilibrium phases such as supersaturated solid solutions, metastable crystalline phases, and metallic glasses (Ref 1, 2). The properties of alloys containing metastable phases are closely related to the morphology and structure of these phases. Hence, for the proper utilization of these materials, their thermal stability is an important criterion. As a result, the main thrust of rapid quenching techniques in the last several years has been structural characterization for the development of microcrystalline alloys with improved properties, especially those based on aluminum (Ref 3). In this connection, a study of the Al-rich end of Al-Ti-Cu and Al-Ti-Ni systems has been undertaken. In this paper attention is focused on the coarsening behavior of the equilibrium phase in two aluminum-base alloy systems as a function of aging times at one temperature, 698 K.

2. Experimental Procedure

Buttons of two Al alloys, one containing (wt%) 6.0Cu-1.0Ti and the other containing 1.97Ni-1.0Ti, were prepared from pure Al, Cu, Ni, and Ti in an argon arc-melting furnace. These compositions were obtained quantitatively by elemental analysis of the buttons using the wavelength dispersive spectroscopy (WDS) technique. The compositions of the Al alloys determined by the WDS technique (wt%) were 5.8Cu-1.1Ti and 2.01Ni-1.15Ti and are within ±5% of the intended compositional values based on the measured amounts of the metals used to make the alloys. Thin foils of the two alloys were given external annealing treatments at 698 K for periods varying from 1 to 465 h after encapsulation under vacuum in quartz tubes to follow structural changes. The foils were then examined in a Hitachi 700-H transmission electron analytical microscope operating at 200 KeV to study the development of the precipitates following annealing to various times. The widths of the precipitates were measured from the transmission electron micrographs. About 100 measurements were made to compute the half-width with 95% confidence level.

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3. Results and Discussion

No new phases were observed in the arc-melted alloys. In the Al-Ti-Cu system, equilibrium phases that are coherent have been detected. Al_3Ti phase was first observed after 100 h of static annealing at 698 K, as shown in Fig. 1(a). The precipitates were elongated in shape. Half-widths of the precipitates were 11, 15, 22, and 32 nm after annealing times of 100, 200, 300, and 475 h, respectively. In the case of the Al-Ti-Ni system, the first signs of the formation of equilibrium Al_3Ni precipitates were observed only after 25 h of static annealing at 698 K, as shown in Fig. 2(a). Elongated precipitates with half-widths 55, 120, and 206 nm were observed after 100, 200, and 300 h, respectively. These particles were not uniformly distributed. In the Al-Ti-Cu system, the equilibrium-strengthening phase is Al_3Ti. Energy dispersive analysis of x-rays (EDAX) results for the precipitates are shown in Table 1 and confirm that the precipitates possess a Al_3Ti structure. Al_3Ti has a tetragonal, $D0_{22}$-type crystal structure (Ref 4), with $a = b = 3.8$ Å and $c = 8.6$ Å. In the Al-Ti-Ni system, the equilibrium-strengthening phase was predominantly Al_3Ni. EDAX results for the precipitates (Table 1) also confirm that the precipitate structure corresponds to that of Al_3Ni. Al_3Ni has an orthorhombic crystal structure and is the prototype of the $D0_{20}$ type (Ref 5, 6) with $a = 6.5$ Å, $b = 7.2$ Å, and $c = 4.7$ Å.

The precipitates in the two systems coarsened at rates that depended on the composition of the alloy system. The precipitate particles in the Al-Ni-Ti system grew more than six times faster than those in the Al-Cu-Ti system. This was determined by measuring the size of the precipitates as the half-width along the minor axis at different annealing times, as shown in Fig. 3 for the two different precipitate types. Each data point of Fig. 3 corresponds to an average of 100 measurements of the particle

Table 1 Composition of the precipitates in the two systems

	Element			
System	Al	Ti	Ni	Cu
Al-Ti-Cu	75.10	24.50	...	0.4
Al-Ni-Ti	74.55	0.75	24.70	...

half-width. Scanning transmission electron microscopy of the coarse precipitates confirmed unequivocally the presence of equilibrium phases in the two systems.

At 698 K, the $D0_{22}$-type Al_3Ti precipitates in the Al-Ti-Cu system coarsened at a rate similar to that for $D0_{23}$-type Al_3Zr equilibrium precipitates of the Al-Zr system studied previously

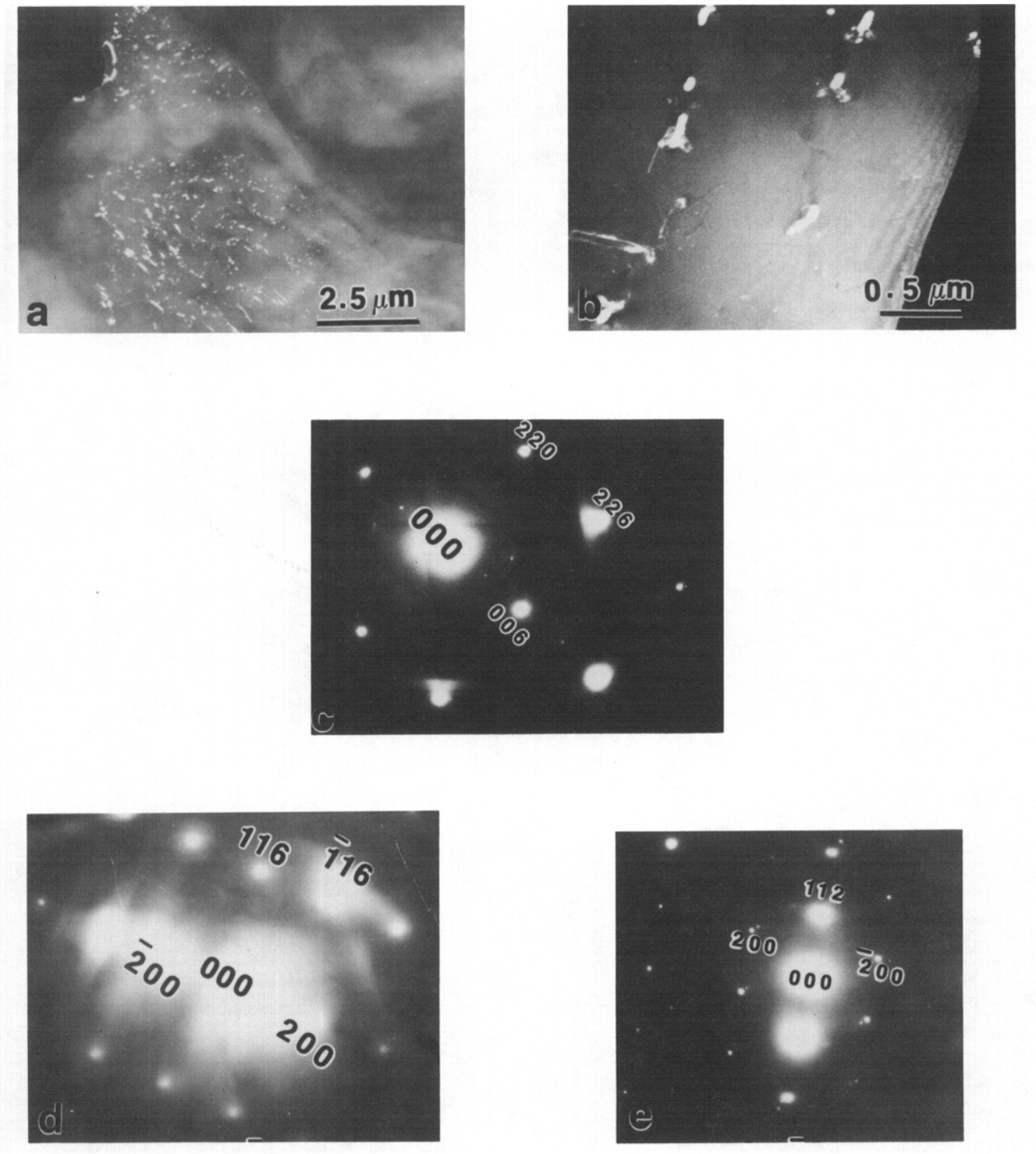

Fig. 1 Transmission electron micrographs for the Al-Ti-Cu system. (a) In dark field using $(200)_{Al_3Ti}$ reflection after 100 h of annealing at 698 K. (b) In dark field using $(200)_{Al_3Ti}$ reflection after 475 h of annealing at 698 K. (c) Diffraction pattern along the $[1\bar{1}0]$ direction for the Al_3Ti precipitate. (d) Diffraction pattern along the $[01\bar{2}]$ direction for the Al_3Ti precipitate. (e) Diffraction pattern along the $[11\bar{2}]$ direction for the Al_3Ti precipitate.

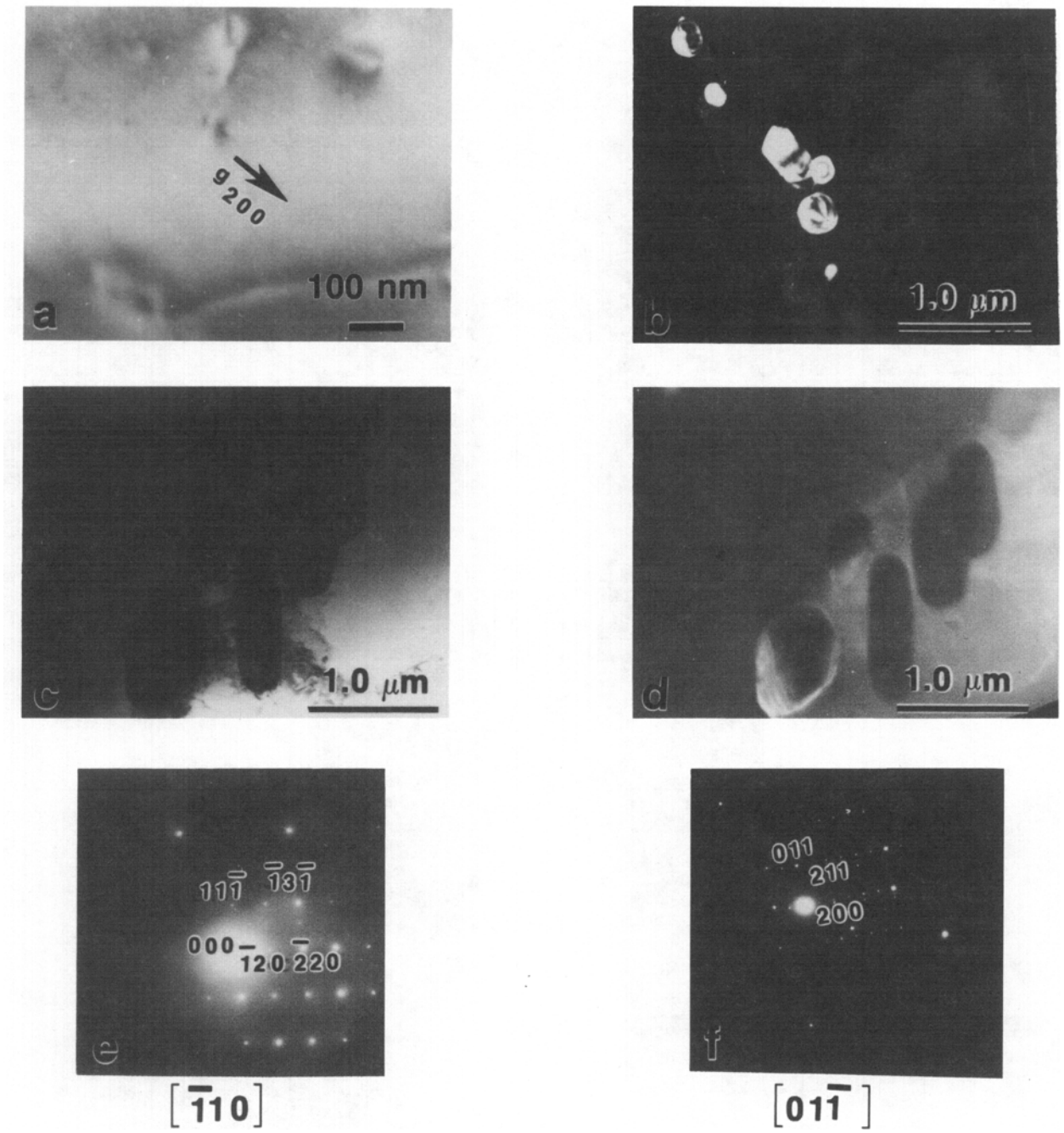

Fig. 2 Transmission electron micrographs for the Al-Ni-Ti system. (a) In bright field after 25 h of annealing at 698 K. (b) In dark field using $(200)_{Al_3Ni}$ reflection after 216 h of annealing at 698 K. (c) In bright field after 300 h of annealing at 698 K. (d) In dark field using $(200)_{Al_3Ni}$ reflection after 300 h of annealing at 698 K. (e) Diffraction pattern along the $[\bar{1}10]$ direction for the Al_3Ni precipitate. (f) Diffraction pattern along the $[01\bar{1}]$ direction for the Al_3Ni precipitate.

(Ref 7), as shown in Fig. 3. It was reported previously (Ref 8) that the ratio of the lattice parameter (a) for intermetallic tetragonal phase Al_3Ti to that for aluminum is 0.95 and matches closely with the a_{Al_3Zr}/a_{Al} ratio of 0.99. Therefore, Al_3Ti and Al_3Zr possess a similar lattice mismatch of 4% with respect to the aluminum matrix and therefore are partially coherent in aluminum. It was also found that they coarsen at similar rates at 698 K. In addition, the Al_3Ti phase is stable as a solid up to 1613 K. It was proposed (Ref 7) that Al_3Zr precipitates coarsen by a volume-diffusion-controlled mechanism. Since the coarsening rate for $D0_{22}$-type Al_3Ti at 698 K is similar to that for the $D0_{23}$-type Al_3Zr, the coarsening of the Al_3Ti would also be rate-controlled by volume diffusion of the solute atoms in aluminum solid solution. Furthermore, it was suggested (Ref 7) that enhanced diffusion due to imperfections could also play a role during high-temperature aging. Because the lattice mismatch of these phases with respect to aluminum is 4%, fully coherent/semicoherent precipitates are rather impossible (Ref 7), as noted in the present work, because of the absence of both an interfacial dislocation network and matrix strain contrast. On

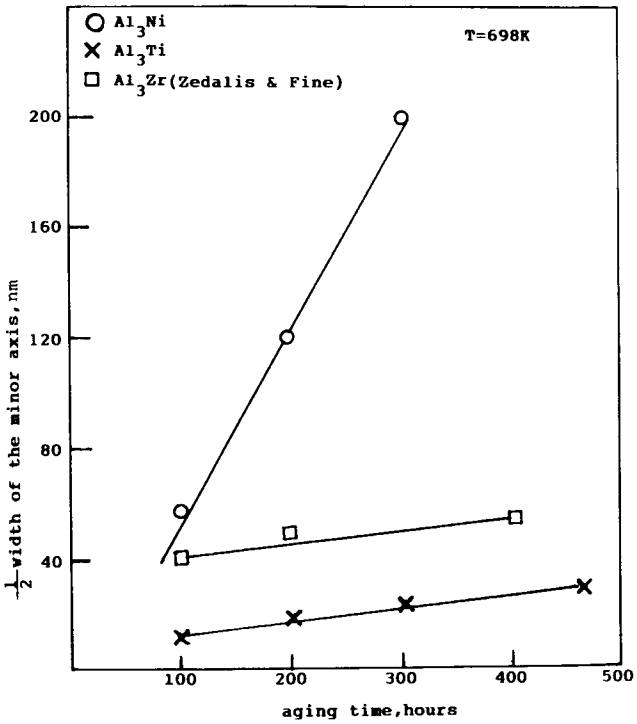

Fig. 3 Plot of the half-width of the precipitate along the minor axis versus aging time for the Al_3Ni, Al_3Ti, and Al_3Zr intermetallics

the other hand, the ratio of the lattice parameter for the Al_3Ni intermetallic (which is stable as a solid up to 1127 K) to that for aluminum is 1.6. These views corroborate the fact that the lattice mismatch and, therefore, the associated interfacial energy with respect to the strengthening phases in the Al-Ni-Ti system are higher than that in the Al-Ti-Cu system. Therefore, Al_3Ni particles coarsened six times faster than Al_3Ti particles, as observed in the present study, probably by a mechanism akin to that for Al_3Ti coarsening. Precipitates associated with higher interfacial energy generally tend to form heterogeneously at grain boundaries where the nucleation is catalyzed due to lower activation energy for nucleation (Ref 8).

In addition, Fig. 1 and 2 suggest that the particles, which were initially spherical, quickly become elongated in shape in both alloy systems after prolonged aging. The aspect ratio of the particles increased with increasing aging times at 698 K. The precipitate particles were often seen to be associated with dislocations, as is evident from Fig. 4. Such connecting dislocations are believed to act as conduits for solute diffusion to assist the coalescence of spherical particles and form elongated precipitates. Numerous models have been proposed to explain the formation of elongated particles. The model of Izumi and Oelschlagel (Ref 9), which considers the formation of elongated particles as a result of coalescence of particles, would be appropriate in the present study. Helical dislocations were not found in these systems, so the model of Ness (Ref 10) is inapplicable.

4. Conclusions

At 698 K:

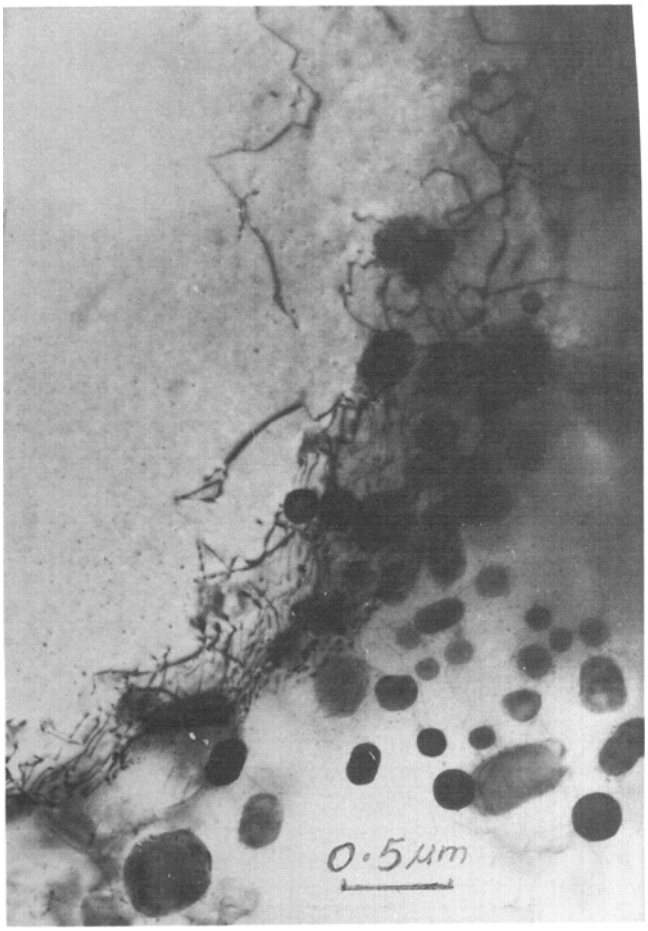

Fig. 4 Transmission electron micrograph of the Al-Ti-Ni alloy annealed for 216 h at 698 K

- The precipitation of partially coherent Al_3Ti and Al_3Ni strengthening phases in aluminum matrix was slow.
- Al_3Ni and Al_3Ti intermetallics became incoherent and elongated with long annealing times.
- The Al_3Ti precipitates coarsened at a rate similar to that of equilibrium Al_3Zr precipitates.
- Al_3Ni precipitates coarsen six times faster than equilibrium Al_3Ti or Al_3Zr precipitates.

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