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Publication Date

1984-07-01

UC-25
LBL-18147
Preprint c. 1



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July 1984

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OBSERVATIONS OF G.P. ZONES AND THEIR TRANSFORMATION
TO γ' IN THE Al-Ag SYSTEM

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Introduction

The precipitation process in the Al-Ag system has been studied extensively by x-ray diffraction and transmission electron microscopy techniques, because this system very nearly represents the ideal case where there is no size difference between the matrix and solute atoms, i.e. the diameters of Al and Ag atoms differ by less than 1%. While the basic aging sequence for an Al-rich alloy quenched from the solid solution α phase and aged within the metastable solvus is generally accepted as:



there is considerable disagreement as to the exact structures of each of these phases and the intermediate stages between them. For example, two models for G.P. zones have been proposed from x-ray small-angle scattering experiments: 1) Guinier and Walker (1) proposed that less than half of the total Ag was contained in spherical, Ag-rich G.P. zones, which were surrounded by Ag-depleted halos, and 2) Baur and Gerold (2) interpreted their results as evidence that all of the Ag was contained within the zones, and that the matrix was at a low, uniform

Ag concentration, i.e. that there is a miscibility gap in the Al-Ag system. Although the latter interpretation is generally accepted as correct, we will also show that in some cases, the former interpretation may also be valid. In addition, while some investigators (3) have proposed that γ' results from growth of the G.P. zones, Nicholson and Nutting (4) and Hren and Thomas (5) have shown conclusively by transmission electron microscopy, that γ' nucleates heterogeneously on both perfect and Frank dislocations. However, our results will again show that in some cases, the former interpretation may also be correct.

Experimental Procedures

An Al-14.92 w/o Ag (4.2 a/o Ag) alloy was vacuum melted and cast, using Al and Ag of 99.99% purity. The ingot was homogenized at 535° C for about 40 hours to reduce segregation, and then hot and cold-rolled to 7mil final thickness. Several pieces of the 7mil sheet were vacuum encapsulated in quartz tubes, solution annealed for 4 hours at 550° C in a vertical furnace, immediately transferred to a horizontal furnace at 350° C where they remained for 12 minutes, and then quenched in cold water, where the encapsulation tubes were broken upon contact with the water. Thin foils were polished using a 25% HNO₃/75% CH₃OH solution at approximately -30° C, 15V and 25mA. Ion-beam milling was also used to heat the foils slightly. Milling was performed for about 5 minutes, using an accelerating voltage of 4keV and 0.3mA total gun current. The foils were examined in a Philips 301 microscope at 100keV.

Results and Discussion

The heat-treatment employed was designed to produce a wide dispersion of plate-shaped γ' precipitates, and this was accomplished. However, an additional spherical-shaped phase was observed to precipitate in the matrix in between the well-developed γ' precipitates after about six months aging at room temperature. This phase is clearly evident in the bright-field micrograph shown in Fig. 1(a). By ion-beam milling, it was possible to partially transform some of the spherical-shaped precipitates into plate-shaped precipitates, several of which are shown in Fig. 1(b).

Diffraction experiments were performed in order to characterize the newly formed precipitates, and these results are shown in Figs. 1(b) through (d). It was possible to distinguish two variants of the γ' plate-shaped precipitates in dark-field by placing an objective aperture around the two strong precipitate reflections near the $\langle 020 \rangle$ matrix spots, shown in Fig. 1(d). These reflections are actually $\langle 10\bar{1}1 \rangle$ streaks from the plate-shaped hcp precipitates, displaced along $\langle 111 \rangle$ directions from the $\langle 020 \rangle$ matrix spots. However, there were no additional spots in the $\langle 001 \rangle$ diffraction pattern due to the spherical precipitates and in fact, it was found that the intensity of these precipitates was largely due to structure factor contrast and could be maximized in dark-field by including diffuse scattering around one of the diffracted beams in the objective aperture. It was therefore concluded that this phase is fcc and coherent with the matrix, typical of G.P. zones, although these G.P. zones are up to 800Å in diameter, a size which is unusually large for such a structure. That the spherical

particles are G.P. zones and not surface deposits is evident from the observation that the precipitates remain spherical, while the plate-shaped γ' precipitates change shape as the foil is tilted from an $\langle 001 \rangle$ zone axis in Fig. 1(b) to an $\langle 112 \rangle$ zone axis in Fig. 2. Although the G.P. zones appear spherical at this level of resolution, it is likely that they are faceted along low-index matrix planes on an atomic level, as recently observed by Gronsky (6).

The overall transformation sequence from G.P. zones to γ' was determined by examining a number of different precipitates in varying stages of transformation, as illustrated by the series of micrographs in Fig. 2. The G.P. zones appear to decompose initially across their diameter, as evidenced by the light globular areas indicated by arrows in (a). Plate-shaped γ' then nucleates in these areas and on the surfaces of the spheres, often growing through their centers and extending beyond their periphery, as shown in (b). The spheres then continue to decompose to form a parallel series of γ' plates of characteristic separation, as shown in (c), and the final product is a set of parallel precipitates whose diameters (or lengths in projection) are still representative of the initial G.P. zone size, as evident in (d). Since G.P. zones nucleate homogeneously in this system (4) this sequence shows that heterogeneously nucleated precipitates such as γ' can nucleate on a homogeneously nucleated predecessor.

Also notice that G.P. zones on the order of 40 to 70 \AA in diameter have nucleated throughout the matrix and that their absence around the larger G.P. zones ($\sim 800\text{\AA}$ in diameter) creates a halo effect, indicating that the larger zones are surrounded by a shell, depleted

in solute. The average width of the depleted layer is about 100\AA . The presence of these small zones also means that four distinctly different precipitates coexist in this sample. The nucleation of γ' precipitate plates directly from G.P. zones, and the Ag-depleted halos around these zones, disagree with some of the more recent theories on this system, as discussed previously.

Conclusions

- 1) Ion-beam milling can induce significant microstructural changes in thin foils of Al alloys if care is not taken to avoid heating during milling.
- 2) Plate-shaped γ' precipitates can nucleate heterogeneously on unusually large, homogeneously nucleated spherical G.P. zones.
- 3) These Ag-rich G.P. zones are surrounded by about a 100\AA layer, depleted in Ag.

Acknowledgements

This work is supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

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Figure Captions

Fig. 1 (a) Bright-field micrograph showing unusually large, spherical G.P. zones in between well-developed γ' precipitates which were produced by the 350°C aging treatment. (b) and (c) Bright-field/dark-field pair showing two variants of γ' precipitates illuminated by placing an objective aperture around the two precipitate reflections near the $\langle 020 \rangle$ matrix spot, shown in (d).

Fig. 2 (a) through (d) Transformation sequence of spherical G.P. zones to plate-shaped γ' precipitates.

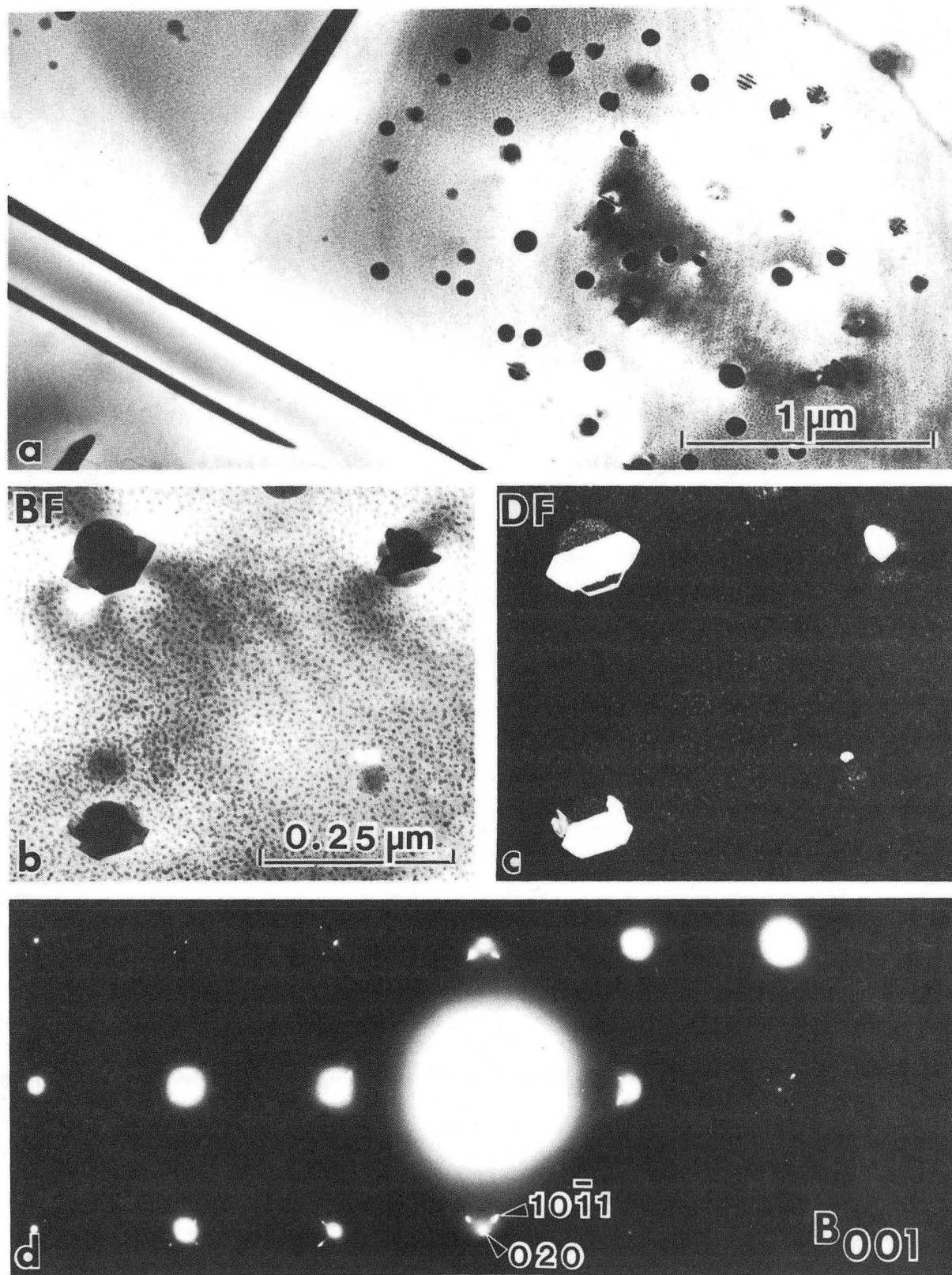


Fig. 1

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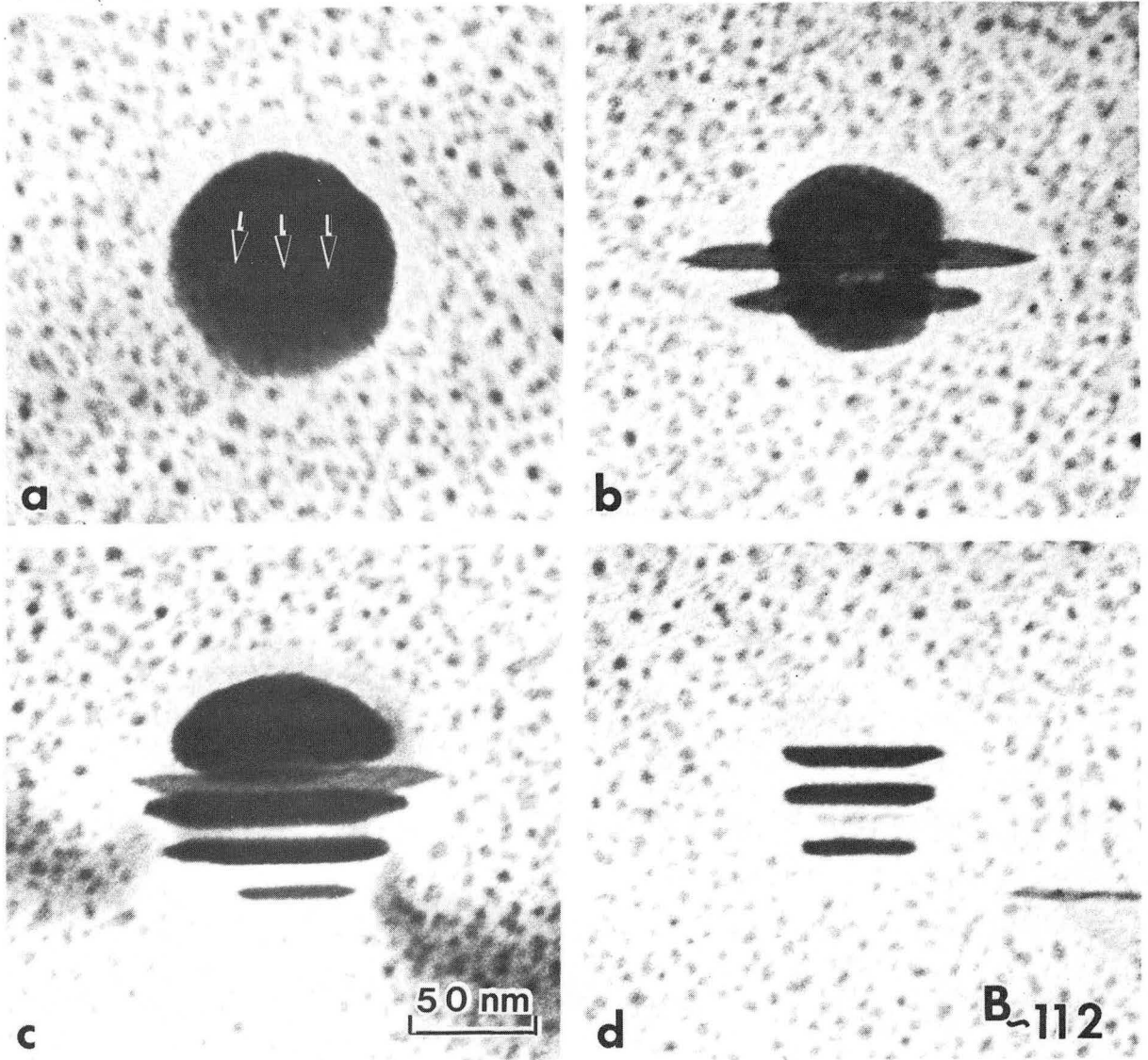


Fig. 2

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