Coarsening of $\beta'$ Precipitates in an Isothermally-Aged Fe$_{75}$-Ni$_{10}$-Al$_{15}$ Alloy

Orlando Soriano-Vargas$^{1,2}$, Maribel L. Saucedo-Muñoz$^1$, Victor M. Lopez-Hirata$^1$ and Ana Ma. Paniagua-Mercado$^1$

$^1$Instituto Politecnico Nacional (ESIQIE), Apartado Postal 118-395, D.F. 07051, Mexico
$^2$UAPT, Universidad Autonoma del Estado de Mexico, Toluca, Estado de Mexico. C.P. 52640, Mexico

The coarsening process of the $\beta'$ precipitates was studied in an isothermally aged Fe$_{75}$-Ni$_{10}$-Al$_{15}$ alloy. The aging treatments at 750, 850, and 920 °C caused the precipitation of the $\beta'$ phase with the B2 type crystalline structure in a bcc ferritic matrix phase. As the aging progressed, the initial rounded shape changed to cuboids aligned in the (100) directions of the ferritic matrix, and finally to rectangular plates also aligned in the same direction. The coarsening process of the $\beta'$ phase was faster as the aging temperature increased. Nevertheless, the highest hardness and slowest overaging process took place during the aging at 920 °C. This increase in hardness seems to be associated with the more rapid formation of the cuboid precipitates aligned in the (100) direction of the ferritic matrix. [doi:10.2320/matertrans.M2009332]

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1. Introduction

The precipitation of the $\beta'$ phase is important for strengthening at high temperatures in different engineering ferritic alloys such as PH stainless steels, nitralloy, Fe-Cr-Ni-Al based alloys, etc. They are used in industrial components which require good mechanical strength and oxidation resistance at high temperatures. The $\beta'$ phase is an ordered phase of the B2 type crystalline structure. The coarsening resistance of precipitates is a key factor to keep the high strength at high temperatures in this type of alloys. An alternative to have a good coarsening resistance, it is a low value of lattice misfit which maintains a coherent interface between precipitate and matrix. Thus, the purpose of present work is to analyze the effect of structural and morphological characteristics of the $\beta'$ precipitates on the coarsening behavior during the isothermal aging of an Fe$_{75}$-Ni$_{10}$-Al$_{15}$ alloy.

2. Experimental Procedure

An Fe$_{75}$-Ni$_{10}$-Al$_{15}$ alloy was melted using pure metallic elements in an electrical furnace under an argon atmosphere. The ingot of $30 \times 10 \times 10$ mm was encapsulated in a quartz tube with gas argon and then homogenized at 1100 °C for one week. Specimens were solution treated at 1100 °C for 1 h and subsequently aged at temperatures of 750, 850 and 920 °C for times from 0.25 to 750 h. The aged specimens were analyzed with a diffractometer using Co Kα radiation. These samples were also observed with a SEM analysis with EDS detector at 20 kV. TEM specimens were prepared by a twin-jet electropolishing method with an electrolyte composed of 25 vol% nitric acid in methanol at $-60$ °C and 10 V (d.c.). Vickers hardness was tested for the aged specimens using a load of 100 g.

3. Results and Discussion

3.1 Microstructural evolution of coarsening

Figure 1 shows the X-ray diffraction (XRD) patterns for the Fe$_{75}$-Ni$_{10}$-Al$_{15}$ alloy solution treated and then aged at 750 °C for 5 h.

![XRD patterns for the Fe$_{75}$-Ni$_{10}$-Al$_{15}$ alloy solution treated and then aged at 750 °C for 5 h.](image)

This is in agreement with the expected one from the equilibrium Fe-Ni-Al diagram. SEM micrographs of precipitates are shown for the sample aged at 750 and 920 °C for different times in Figs. 2(a)–(c) and (d)–(f), respectively. The shape of the $\beta'$ precipitates was rounded particles without any preferential alignment for the aging at 750 °C up to 75 h and 920 °C up to 0.5 h, Fig. 2(a). A further aging changed the shape of the $\beta'$ precipitates to cuboids with a preferential alignment on the (100) directions of the ferritic $\alpha$ phase, Figs. 2(c)–(e). This alignment can be
Fig. 2  SEM micrographs for the Fe$_{75}$-Ni$_{10}$-Al$_{15}$ alloy aged at 750°C for (a) 75, (b) 250, and (c) 500 h, and at 920°C for (d) 25, (e) 100 and (f) 200 h.
clearly observed in a dark field TEM micrograph of the sample aged at 850 °C for 150 h and its corresponding electron diffraction pattern with a zone axis [100] shown in Fig. 3. A prolonged aging at 900 °C promoted the change of shape to rectangular plates also aligned in the (100) directions, Fig. 2(f). The volume percentage of precipitation was determined to be about 30, 25 and 20% for the samples aged at 850 and 920 °C for 500 h and at 920 °C for 200 h, respectively.

3.2 Growth kinetics of coarsening

The variation of the \( \beta^- \) precipitates size expressed as \( r^3 - r_o^3 \) with aging time for the sample aged at 750, 850 and 920 °C is shown in Fig. 4. It can be noticed that the experimental data fit to a straight line for each temperature. Thus the growth kinetics of coarsening followed the behavior predicted by the Lifshitz-Slyozov-Wagner (LSW) theory for coarsening controlled by volume diffusion. This fact shows a good agreement with the modified theory for the diffusion-controlled coarsening in ternary alloys, which predicts that growth kinetics is similar to that of LSW theory. The size distribution of precipitates is shown in Figs. 5(a)–(c) for the sample aged at 750, 850 and 920 °C for 200 h, respectively. The probability density \( \rho^3(h) \) was determined with the following equation:

\[
\rho^3(h) = k \cdot \frac{1}{1 + (h/k)^{1/2}}
\]

where \( k \) is the coarsening rate constant.

![Fig. 3 DF-TEM micrograph and electron diffraction pattern for Fe\(_{75}\)-Ni\(_{10}\)-Al\(_{15}\) alloy aged at 850 °C for 150 h.](image)

![Fig. 4 Plot of \( r^3 - r_o^3 \) vs. aging time for the Fe\(_{75}\)-Ni\(_{10}\)-Al\(_{15}\) alloy aged at 750, 850 and 920 °C.](image)

![Fig. 5 Size distribution of precipitates for the Fe\(_{75}\)-Ni\(_{10}\)-Al\(_{15}\) alloy aged at (a) 750, (b) 850 and (c) 920 °C for 200 h.](image)
3.3 Precipitation hardening behavior

Equation (2) represents the number of particles in a given class interval \( \Delta r \). The normalized radii is defined as the ratio of \( r/\Delta r \).

\[
\rho^2 f(\rho) = \frac{N(r, r + \Delta r)}{\sum N(r, r + \Delta r) \Delta r} \frac{r}{\Delta r}
\]

where \( \rho \) is the average radius of the particle and \( N(r) \) represents the number of particles in a given class interval \( \Delta r \). The normalized radii is defined as the ratio of \( r/\Delta r \). It can be seen that the size distribution is broader and lower than that predicted by the LSW theory because of the high volume fraction of precipitates, which has been reported in the coarsening process of several alloy systems.\(^8\)\(^-\)\(^12\) It has been observed\(^13\),\(^14\) that the growth or shrinkage rate of an individual particle depends not only on its normalized radii but also on its local environment. That is, a particle surrounded by several larger particles will grow slower, or shrink faster, than a particle of the same size whose neighbours are small. Thus, as the volume fraction increased, the particle size distribution widened and the coarsening rate also increased.

It was also observed that the higher aging temperature, the faster coarsening kinetics of the \( \beta' \) precipitates because of the increase in volume diffusion.\(^13\),\(^15\)

The chemical composition of the \( \beta' \) precipitates is shown in Table 1 for the samples aged at 750 and 920 °C for 200 h. The chemical composition of \( \beta' \) precipitates is close to the expected composition of Fe(NiAl) compound in the case of the sample aged at 850 °C. In contrast, the composition of \( \beta' \) precipitates becomes richer in Fe with almost the same proportion of Ni and Al in the sample aged at 920 °C. The XRD results showed that the crystalline structure for the \( \beta' \) precipitates in both aged samples corresponded to the B2 type. Table 2 shows the lattice parameter of \( \beta' \) precipitates and the ferritic matrix determined from the XRD patterns for the samples aged 850, 900 and 920 °C for 0.25 h. The lattice parameter was determined with a precision of ±0.0001 nm. Table 2 also shows the lattice misfit, \( \delta \), between the matrix and precipitates and it can be noticed that the lattice misfit increases with increasing aging temperature. It is well known\(^6\),\(^13\),\(^16\),\(^17\) that the elastic-strain energy is proportional to the lattice misfit \( \delta \) and it affects the morphology and alignment of the precipitated phases. The higher \( \delta \) value for aging at 920 °C could explain the fastest formation of cuboids and its corresponding alignment in the (100) direction, elastically softer direction for the ferritic matrix.

4. Conclusions

The aging process of the Fe\(_{75}\)-Ni\(_{10}\)-Al\(_{15}\) alloy promoted the precipitation of the \( \beta' \) (Fe(NiAl)) precipitates with the B2 type crystalline structure. The morphology of \( \beta' \) precipitates was rounded at the early stages of aging and then it changed to cuboids aligned in the (100) directions of the ferritic matrix. A prolonged aging caused the formation of rectangular plates also aligned in this direction. The coarsening process followed the growth kinetics predicted by the LSW theory. Nevertheless, the hardness peak was higher and the overaging process occurred later in the sample aged at 920 °C than those of sample aged at 750 °C. This behavior can be attributed to the fast formation of cuboid morphology and alignment in the (100) direction due to the higher lattice misfit between the ferritic matrix and \( \beta' \) precipitate at this aging temperature.
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REFERENCES