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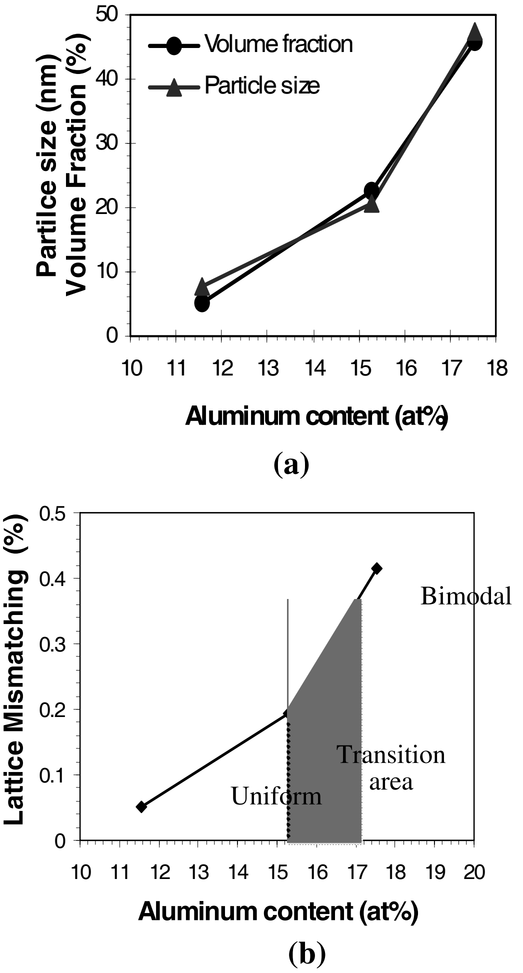

Fig. 2. Particle size and volume fraction of γ′ (a), and (b) γ-γ′constrained lattice mismatch as a function of aluminium content. Samples were solutioned at 1250°C for 45 min and then quenched in ice brine.

with an equivalent mean diameter about 100 nm and fine spherical precipitates with an average size of about 15 nm. It seems that the formation of fine spherical precipitates during rapid quenching occurs simultaneously with the morphological transition of large precipitates. There for, in the composition range between 15.3 and 17.5 at.%Al, and accordingly the range of γ-γ′ lattice mismatch between 0.20 and 0.4% - Fig. 2(b) - there is a transition from uniform to bimodal distributions of both the size and the morphology of γ′ precipitates. As mentioned above, while there is evidence that the transition begins in the Ni-15.3 at.%Al alloy, the present data do not provide an evidence for a definite determination of the end of this transition. In addition, the bimodal size and morphological distribution of γ′ indicates that precipitation in high Al-content alloys during rapid quenching occurs in two detached temperature regimes with no precipitation between them. Large cuboidal precipitates nucleate in higher temperature range and fine spherical precipitates nucleate in the lower temperature range.

4. Summary

The effect of aluminium content of the Ni-Al binary alloys on the precipitation of γ′ during rapid quenching from the solution temperature was investigated using electron microscopy and XRD techniques. According to the results, in low Al-content alloy, the phase separation and ordering of the γ′ begin simultaneously during rapid quenching through continuous nucleation and growth. With increasing Al content, in addition to the volume fraction and size of precipitates, the coherency strains between γ and γ′ increases. In the composition range between 15.3 and 17.5 at.%Al, and accordingly the range of γ-γ′ lattice mismatch between 0.20 and 0.4%, a transition from uniform spherical precipitates to bimodal spherical-cuboidal precipitates occurs. Fine spherical precipitation occurs simultaneously with morphological transition of large precipitates to cubic shape. Discontinuous size distribution of the γ′ particles in bimodal microstructure of the Ni-17.5 at.%Al alloy indicates that the precipitation in the high Al-content alloys occurs in two detached temperature regimes with no precipitation between them. Large cuboidal precipitates form at higher temperature range while the fine spherical precipitates nucleate at lower temperatures.

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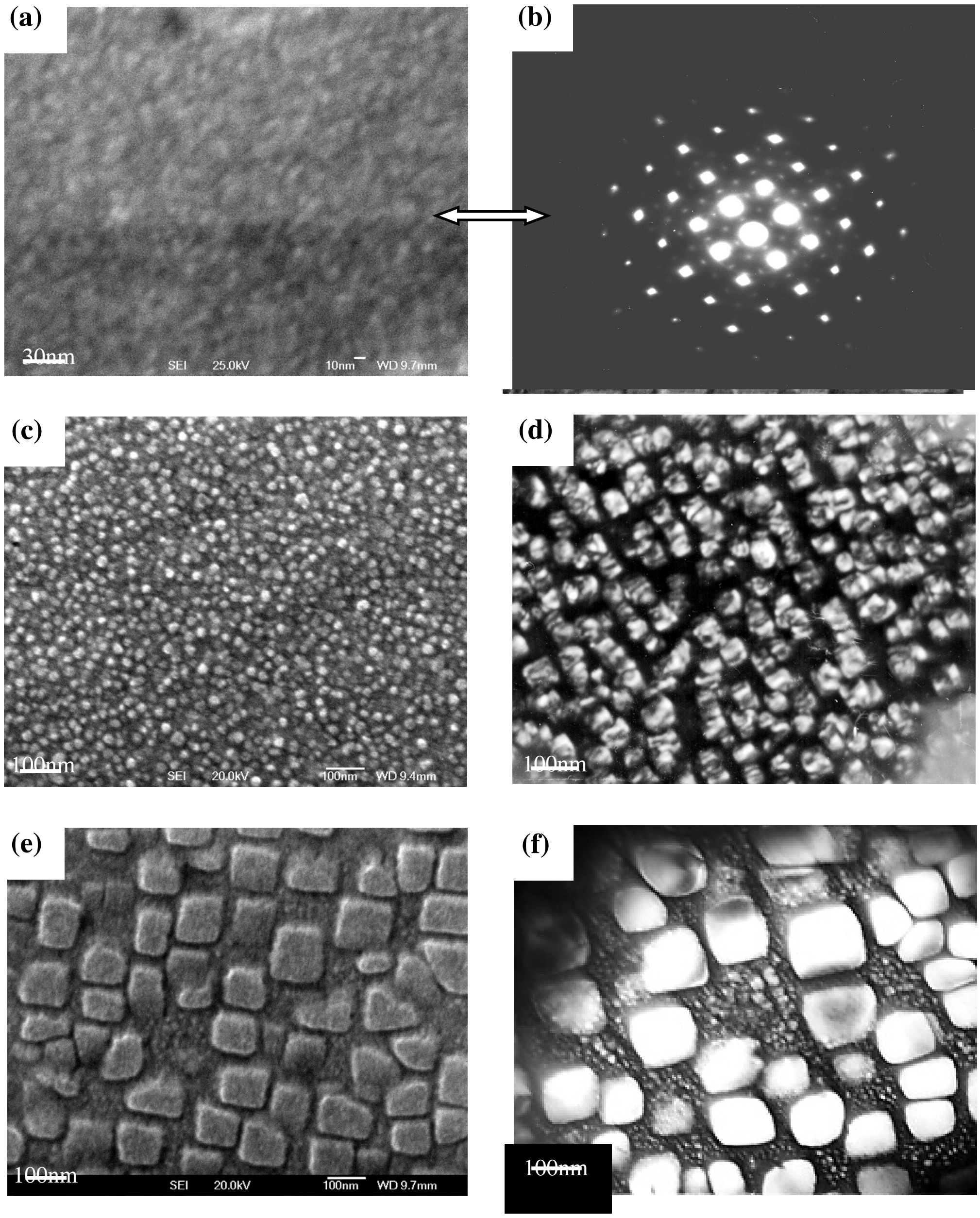

Fig. 1. FEGSEM (left hand) and dark field TEM (right hand) micrographs of solutioned and quenched samples; (a) Ni-11.56at%Al; (b) Selected area electron diffraction pattern of Ni-11.6 at.%Al alloy. The foil was oriented in <001> direction and the superlattice reflections (weak points) are {100} and {110} type. (c,d) Ni-15.3 at.%Al; (e,f) Ni-17.5 at.%Al.

temperature range for precipitation of γ' in Ni-17.5 at.%Al alloy, which leads to a bimodal distribution. This is also illustrated in Fig. 1(e,f), showing a mixture of large cuboidal particles

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microstructure of the Ni-11.56 at.%Al sample in Fig. 1(a) and its related electron diffraction pattern in Fig. 1(b) suggest, in agreement with the literature [17] - [21], that nucleation and growth are the prevailing mechanism. In consistence with the findings of Schmuck et al. [12], these figures imply that ordering and phase separation take place simultaneously during rapid quenching. The result show clearly that precipitation of γ′, even in the dilute Ni-Al alloys, starts during quenching. Moreover, the precipitation appears to occur through fluctuations, small in degree but large in extent, resembling uniform homogeneous nucleation. This indicates an infinitesimally small activation energy for nucleation in this alloy.

As shown in Fig. 1(c-f) a transition in morphology, size and distribution is occurred with increasing the Al content. The uniform spherical particles in Ni-15.3 at.%Al alloy change to a bimodal distribution of fine-spherical and large-cuboidal precipitates in the Ni-17.5 at.%Al alloy. In addition to the particle size and volume fraction of γ′ – Fig. 2(a) – the γ-γ′ lattice mismatch as shown in Fig. 2(b) also increases with increasing the Al content. This also indicates that the coarsening rate of the γ′ precipitates increases with increasing the γ-γ′ lattice mismatch. Figure 2 also shows that Al content may have an effect on the γ-γ′ lattice mismatch, without changing the shape of precipitates. According to Qiu [5] and Conley et al. [7] when the lattice mismatch is low, the coarsening of the precipitates is achieved without changing their morphology. However, by further increasing the Al content ,the change in lattice mismatch is associated with the morphological changes. The morphological development of γ′ via lattice mismatch is qualitatively in agreement with that of Refs. [13], [22] - [25]. However, the value of lattice mismatch for the cubic γ′ as obtained in this work is somewhat lower than that reported by Stoloff [22] and Borodians'ka [25] and their co-workers, who attributed spherical, cuboidal and platelet morphologies of the γ′ precipitates to the 0-0.2%, 0.5-1.0% and higher than 1.25% mismatch values, respectively. This discrepancy can be related to the fine spherical precipitates in Ni-17.5 at.%Al alloy, as is also evident by the results of X-ray diffraction experiments.

In the Ni-15.3 at.%Al alloy, possibly due to the lower supersaturation of the solid solution, both the nucleation temperature of precipitates and the temperature range for precipitation is too low to form a bimodal distribution of precipitates. Therefore, apart from a few extremely small precipitates within the region between large spherical particles, Fig. 1(c), a uniform distribution with an average particle diameter of about 20 nm is formed. However, TEM of the Ni-15.3 at.%Al alloy - Fig. 1(d) – gives an indication of the threshold of the transition from uniform to bimodal distribution in this sample. By increasing the Al content of alloys, not only the decomposition temperature increases, but also the required undercooling for decomposition reaction $\gamma \rightarrow \gamma' + \gamma$ decreases [26]. Thus, there will be a wide

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precipitates, and lacked a discussion on the role of Al content on γ-γ′ lattice mismatch and morphological evolution. Lund et al. [16] studied a wide range of Ni-Al binary alloys, focusing on the effect of elastic stress on the coarsening kinetics. However, this study did not consider initial stages of precipitation, nor did it discuss the morphology of the precipitates.

Generally, previous studies made use of indirect techniques, e.g. based on a measurement of the variation of physical or mechanical properties of the samples, in order to characterize early stages of γ′ precipitation. The present study aims to complement these studies, by making use of direct microscopic observations, which provide information on the size, morphology and distribution of the precipitates. Specifically, this study focuses on the role of Al content in the early stages of precipitation in Ni-Al binary alloys, by means of rapid quenching. In addition to microstructural analysis, an estimate of γ-γ′ lattice mismatch was obtained from X-ray diffraction (XRD) patterns.

2. Experimental Procedure

Three compositions of Ni-Al binary alloys, containing 11.6, 15.3 and 17.5 at.%Al were cast using 99.99 % pure metals in an argon protected arc-melting furnace. Each specimen was re-melted several times to ensure compositional homogenity. The cast specimens were homogenized inside the evacuated quartz tubes at 1200°C for 30 hours followed by 45 minutes full solutionizing at 1250°C and then were drop quenched into iced brine. The average size and volume fractions of the γ′ precipitates in as quenched samples were determined through image analysis of micrographs. For cubic particles, the diameter of an equivalent spherical particle was considered as particle size.

Microstructural studies were carried out using a JEOL model 7000 field emission gun scanning electron microscope (FEGSEM) and PHILLIPS model CM20 transmission electron microscope (TEM) operating at 200 keV. The SEM samples were mechanically ground and finally polished down by 0.04 μm colloidal silica. Then they were chemically etched for long times in a solution consisting of nitric acid, glacial acetic acid and distilled water in the ratio 2:1:1. Thin foils TEM samples were electro-polished at 15V/75mA in ethanol containing 10% perchloric acid at about -20°C. TEM imaging was carried out using dark field mode.

X-ray diffraction (XRD) experiments were carried out on bulk samples at room temperature using a computer controlled diffractometer, with unfiltered Cu Kα radiation generated at 40 kV and 40 mA. All scans were performed using 0.02-deg step interval and a minimum counting time 0.5 second at each step. The overlapped γ and γ′ peaks were analyzed, as described in details by Kamara et al. [2] using a computer program called "*X'pert*". The constrained lattice mismatching factors, $\varepsilon = (a_{\gamma'} - a_{\gamma})/a_{\gamma}$, were calculated using lattice parameters measured from the XRD patterns, where ε, a_{γ} and $a_{\gamma'}$ are the constraint lattice misfit and lattice constants of γ and γ′, respectively.

3. Results and Discussion

A progressive evolution in the size and morphology of the γ′ precipitates via increasing Al content in the as quenched samples is shown in Fig. 1. The "mottled"

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Effect of Composition on the Precipitation of γ′ in Rapidly Quenched Ni-Al Binary Alloys

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Abstract
The effect of aluminium content on the precipitation of γ′ in rapidly quenched Ni-Al binary alloys was investigated. Microstructural analyses of annealed and quenched samples indicate a transition from uniform to bimodal γ′ phase distribution in the composition range between 15.3 and 17.5 at.%Al. This transition is accompanied by changes in the morphology of γ′ precipitates. The microstructural observations are discussed in view of both kinetic and crystallographic considerations.

Keywords: Ni-Al System, Heat Treatment, γ′ Precipitation, Lattice Mismatch

1. Introduction

The precipitation of $L1_2$ type ordered phase γ′ as the major strengthening mechanism of nickel-base superalloys, has been widely studied in the literature. The chemical composition is one of the most important factors influencing the initial precipitation of γ′ and consequently final microstructure and mechanical properties of the nickel-base superalloys. It influences not only the coherency between γ and γ,′ and consequently γ′ morphology, but also the coarsening rate of the γ′ [1] - [7]. Even though, several studies have concentrated on the effect of alloying elements on γ′ precipitation [3] - [14], there has been relatively little attention to the effect of Al concentration, as a fundamental γ′ forming element, on the precipitation of γ′, especially during rapid quenching. Gentry and Fine [15] studied the initial stages of the precipitation in two different Al contents of Ni-Al binary alloys after short time-low temperature aging. They believed that via increasing the Al content of alloys the decomposition mechanism of the supersaturated γ is changed from nucleation and growth to the spinodal decomposition. However, they judged only by measuring the variation of magnetic properties without a detailed microstructural analysis. Kamara et al. [2] reviewed the literature and plotted the variation of the room temperature lattice parameters of γ and γ′ vs. Al content of the Ni-Al alloys. However, their review focused only on fully coarsened

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