

The influence of ageing on the stability of β - γ/γ' derived microstructures in Ni–Al–Cr–(Co) alloys

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Multiphase Ni–Al–(Fe)–(Cr)–(Co)-based intermetallics with β (B2)– γ (A1)/ γ' (L1₂), β - γ or β - γ' microstructures can exhibit significant room-temperature tensile ductility. In the case of Ni–Al–Cr-based alloys, microstructural development is complicated by the precipitation of α -Cr, which can supplant the γ -phase during ageing of three-phase β - γ/γ' microstructures. An investigation of the stability, during ageing, of cast Ni–Al–Cr–(Co) alloys with microstructures derived from β - γ/γ' is reported. In the as-cast condition, the materials investigated consisted of a dendritic matrix containing L1₀ type martensite and interdendritic γ/γ' . Extensive intra- and interdendritic α -Cr precipitation was also observed. The stability during ageing of the interdendritic γ/γ' microstructure is also considered and transformation of the L1₀ martensite is examined.

1. Introduction

The poor room-temperature ductility and toughness of the B2 type intermetallic compound NiAl (β -phase) has encouraged interest in the addition of ductilizing second-phase regions to a β -phase matrix (e.g. [1–7]). Microstructures composed of a β -matrix plus two-phase γ (A1)/ γ' (L1₂) regions can offer significant tensile ductilities. Such microstructures can be formed in Ni–Al-based intermetallics with additions of either chromium or iron (e.g. [2, 3]). Reliable formation of a β - γ/γ' microstructure is offered by a cast Ni–20 at % Al–30 at % Fe (e.g. [5–7]) alloy. However, the formation of similar microstructures in a relatively aluminium-rich Ni–Al–Cr–(Co) material would be attractive from the standpoint of environmental resistance.

Previous work on microstructural stability in Ni–Al–Cr-based materials with β - γ/γ' -derived microstructures in the as-cast condition has presented somewhat contradictory results. Yang *et al.* [8] observed that an Ni–18 at % Al–9 at % Ti–10 at % Cr alloy initially solidified to a microstructure comprised of β dendrites (with some intradendritic α -Cr) and two-phase γ/γ' interdendritic regions. In the as-cast condition, the γ -phase was stabilized by segregation of chromium from the β -phase to the interdendritic region during solidification. However, Yang *et al.* found that the chromium-rich γ -phase served as a site for the preferential nucleation and growth of α -Cr during short-term (1 min to 24 h) ageing at temperatures of 900 and 1050 °C. This α -Cr precipitation resulted in removal of the γ -phase. Furthermore, in the case of the Ni–Al–Cr ternary system, the available phase diagrams contain a three-phase $\beta + \gamma + \gamma'$ phase field in 1025 and 1150 °C isotherms, but not in the 850 °C isotherm [9].

Behaviour contrasting to that observed by Yang *et al.* was noted by one of the authors [10, 11] for an Ni–23 at % Al–8 at % Cr–11 at % Co–4 at % Ti–1 at % Mo–1 at % V–0.3 at % W–1.3 at % C alloy (whose composition was originally selected to examine microstructural development in aluminide diffusion coatings). In the as-cast condition, this material consisted of dendrites with an L1₀ martensitic matrix and two-phase γ/γ' interdendritic regions (plus a single-phase γ' layer around the dendrites). It was observed that the γ/γ' regions survived ageing at both 950 °C for 140 h and 1100 °C for 170 h (ageing also served to transform the L1₀ martensite back to the B2 phase).

The present investigation considers the microstructural stability of cast Ni–24 at % Al–8 at % Cr–12 at % Co, Ni–24 at % Al–15 at % Cr and Ni–24 at % Al–21 at % Cr alloys during 140 h ageing treatments at 850 and 1100 °C. In the as-arc-melted and chill-cast condition, the authors have observed previously [12] that these materials have microstructures comprised of a dendritic matrix which had transformed to L1₀ martensite and interdendritic regions containing cuboidal γ' in γ . The present work was motivated by the apparently contradictory previous observations (detailed above) of the stability of β - γ/γ' -based microstructures in complex cast and aged Ni–Al–Cr-based alloys containing, for example, γ' stabilizers such as titanium [8, 10, 11]. The present paper focuses on relatively simple Ni–Al–Cr–(Co)-based cast and aged materials without the presence of potentially complicating compositional factors, such as γ' stabilizers. The intention of this work was to examine the influence of chromium (and cobalt) on microstructural stability in these Ni–Al–Cr–(Co) alloys. Hence,

wrought materials and mechanical properties were not considered.

2. Experimental procedure

Ni-24 at % Al-8 at % Cr-12 at % Co, Ni-24 at % Al-15 at % Cr and Ni-24 at % Al-21 at % Cr alloys prepared [12] by arc-melting and chill-casting were aged at a temperature of 850 °C for 140 h and subsequently furnace cooled to room temperature. Based on the results of the 850 °C ageing trials, the Ni-24 at % Al-21 at % Cr alloy was selected for further examination. The as-cast Ni-24 at % Al-21 at % Cr alloy was subjected to a 140 h/1100 °C ageing treatment which was followed by furnace cooling to room temperature.

Metallographic specimens for light microscopy (LM) and scanning electron microscopy (SEM) were prepared from as-cast and heat-treated samples by electrolytic etching at 3 V in a solution comprised of 70 vol % distilled water, 10 vol % glycerol, 15 vol % hydrochloric acid and 5 vol % nitric acid. Transmission electron microscopy (TEM) specimens were prepared by electropolishing using a 30% solution of perchloric acid in methanol at around 35 mA, 12 V and a temperature of approximately -35 °C. Where problems with differential polishing were observed, electropolishing was supplemented by argon-ion milling using dual guns operated at 5 kV with a current of 500 μ A per gun and at gun to specimen angles of 13°–15°.

TEM investigations were conducted using a JEOL 1200 EX instrument operated at 120 kV. SEM studies were undertaken using a JEOL 840 microscope operated at 20 kV. TEM and SEM-based energy dispersive X-ray spectroscopy (EDS) investigations were performed using ultra-thin window (UTW) detectors and a Tracor Northern 5500 analyser.

Ageing of the Ni-24 at % Al-8 at % Cr-12 at % Co alloy was examined using *in situ* heating. This work employed a Philips CM 30 TEM operated at an accelerating voltage of 300 kV, together with a Gatan heating stage. *In situ* observations were conducted in the approximate temperature range 500–1000 °C on samples initially in the as-cast condition.

3. Results and discussion

The as-cast microstructures of the three Ni-Al-Cr-(Co) materials studied in the present work have been discussed in detail elsewhere [12] and will only be summarized briefly here. The bulk of this section will then discuss the influence of ageing on the stability of the initial as-cast microstructures. As-cast microstructures and the changes observed in these microstructures during ageing are summarized in Table I and Fig. 1 for the three alloys examined.

3.1. As-cast condition

The as-cast Ni-24 at % Al-21 at % Cr alloy (Fig. 2) was found to contain dendrites with matrices com-

posed of L1₀ martensite. No evidence was found of the formation of 7R or other phases within the L1₀ martensite. Retained β was not observed. Extensive intradendritic precipitation of α -Cr as fine (10–30 nm diameter) spheres was observed. Coarse rod-like α -Cr precipitates (typically around 40 μ m long and 5 μ m diameter) were also observed intra- and interdendritically (and also crossing dendrite boundaries). A layer of single phase γ' with a thickness of around 1 μ m was noted around the dendrites. The interdendritic regions were strongly chromium-enriched compared to the dendrite interiors. This chromium enrichment correlated with the formation of interdendritic regions with a γ' matrix. The interdendritic γ matrix contained extensive precipitation of γ' cuboids (around 500 nm on-edge).

The as-cast Ni-24 at % Al-15 at % Cr alloy was generally similar to the Ni-24 at % Al-21 at % Cr material. However, unlike the 21 at % Cr material, fine spherical intradendritic α -Cr was not observed in the 15 at % Cr alloy. When compared to that observed in the 21 at % Cr alloy, a relatively low level of γ -stabilizing chromium was present in the interdendritic regions of the 15 at % Cr material. This difference in interdendritic chromium content correlated with the formation of a relatively prominent single-phase γ' layer in the 15 at % Cr alloy compared with the 21 at % Cr material.

The Ni-24 at % Al-8 at % Cr-12 at % Co alloy possessed the same overall (intradendritic L1₀ martensite plus interdendritic γ/γ') as-cast microstructure as the Ni-Al-Cr materials. However, in comparison with the Ni-Al-Cr alloys, the formation of α -Cr was relatively uncommon in the cobalt-bearing material. The Ni-24 at % Al-8 at % Cr-12 at % Co alloy was found to have a relatively prominent interdendritic region. Compared with the cobalt-free alloys, the Ni-Al-Cr-Co material showed relatively little γ' precipitation and the interdendritic region was dominated by the γ -phase. Although some relatively coarse (around 500 nm on-edge) cuboidal γ' was observed in the cobalt-bearing alloy, significant parts of the interdendritic region contained only fine (5–10 nm diameter) spherical γ' . Furthermore, no evidence was found for the formation of a single-phase γ' layer around the dendrites of the Ni-Al-Cr-Co alloy. The relative lack of γ' in the interdendritic microstructure of the as-cast cobalt-bearing alloy correlated with strong enrichment of the interdendritic regions with cobalt and chromium (both of which are γ -stabilizers [13]).

3.2. Aged microstructures

The three materials investigated were found to show significantly different responses to ageing (particularly with respect to the stability of L1₀ martensite, Fig. 3, and γ' formation, Fig. 4), despite the general similarity of their as-cast microstructures. The influence of 850 °C ageing on the microstructural stability of the three Ni-Al-Cr-(Co) alloys will first be considered.

The Ni-24 at % Al-15 at % Cr alloy showed a marked response to ageing. First, consider the

TABLE 1 Summary of as-cast and 140 h at 850°C aged microstructures

Condition	Dendrite matrix	Spherical intradendritic α -Cr	Intradendritic γ' precipitates	Inter- and intra-dendritic α -Cr rods	γ' layer around dendrites	Interdendritic matrix	Co-rich interdendritic α -Cr precipitates
Ni-24 at % Al-21 at % Cr as-cast	L ₁₀ Martensite	Present	Not present	Present	Present	Monomodal cuboidal γ' in γ	Not present
Ni-24 at % Al-21 at % Cr aged	Predominantly L ₁₀ martensite with some β -phase	Present	Present in β -phase regions only	Present	Present	Bimodal coarse cuboidal plus fine spheroidal γ' in γ	Not present
Ni-24 at % Al-15 at % Cr as-cast	L ₁₀ martensite	Not present	Not present	Present	Present	Monomodal cuboidal γ' in γ	Not present
Ni-24 at % Al-15 at % Cr aged	β -phase	Not present	Present	Present	Not distinct from interdendritic matrix	Single phase γ'	Not present
Ni-24 at % Al-8 at % Cr-12 at % Co as-cast	L ₁₀ martensite	Present	Not present	Occasional precipitates present	Not present	Predominantly monomodal spheroidal γ' in γ	Not present
Ni-24 at % Al-8 at % Cr-12 at % Co aged	β -phase	Present	Present	Occasional precipitates present	Not distinct from interdendritic matrix	Single-phase γ'	Present

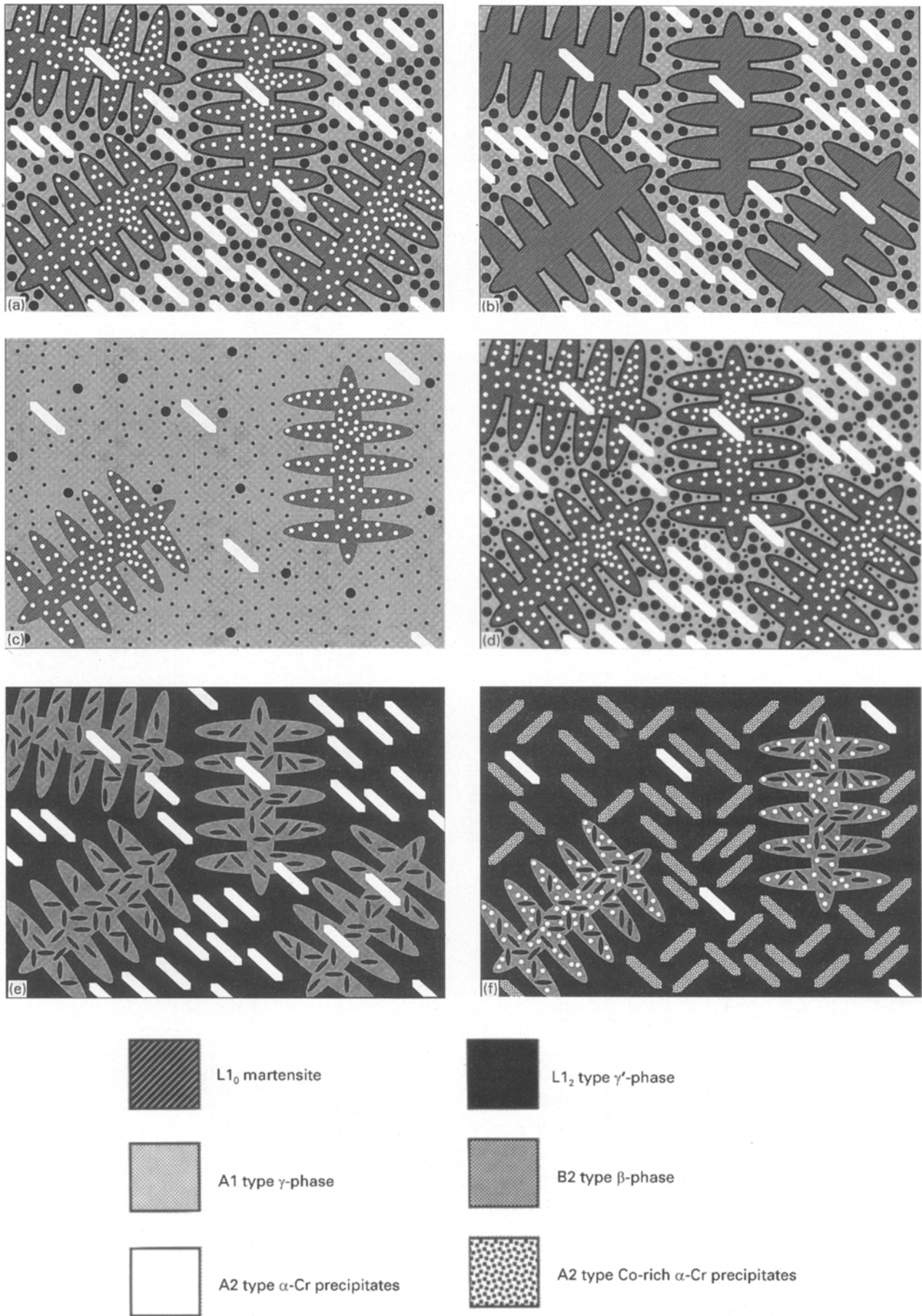


Figure 1 Schematic illustration of as-cast and 140 h at 850 °C aged microstructures. (a) Ni-24 at % Al-21 at % Cr, as-cast; (b) Ni-24 at % Al-15 at % Cr, as-cast; (c) Ni-24 at % Al-8 at % Cr-12 at % Co, as-cast; (d) Ni-24 at % Al-21 at % Cr, aged; (e) Ni-24 at % Al-15 at % Cr, aged; (f) Ni-24 at % Al-8 at % Cr-12 at % Co, aged.

intradendritic regions. The 140 h/850 °C ageing treatment was found completely to remove the L1₀ martensitic phase from the dendrite interiors. After ageing, the dendrites were found to possess a β-phase matrix containing extensive γ' precipitation. The γ' precipitates were found to be roughly ellipsoidal in shape with lengths typically in the range 500 nm to 1 μm and widths of around 200–400 nm. An orientation relationship was observed between the β and γ' phases

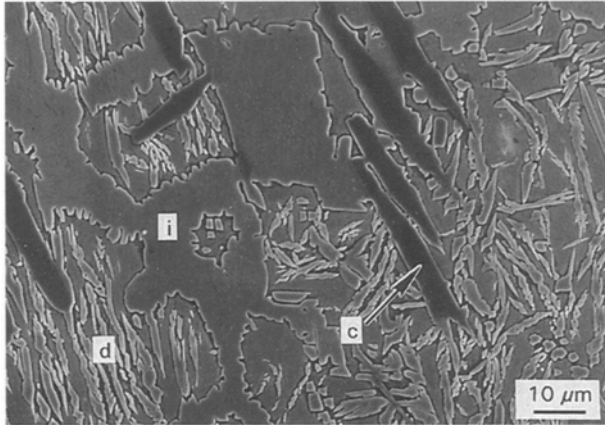


Figure 2 Secondary electron image of as-cast Ni-24 at % Al-21 at % Cr (d = L1₀ type martensitic dendrite interior, i = interdendritic γ/γ', c = α-Cr).

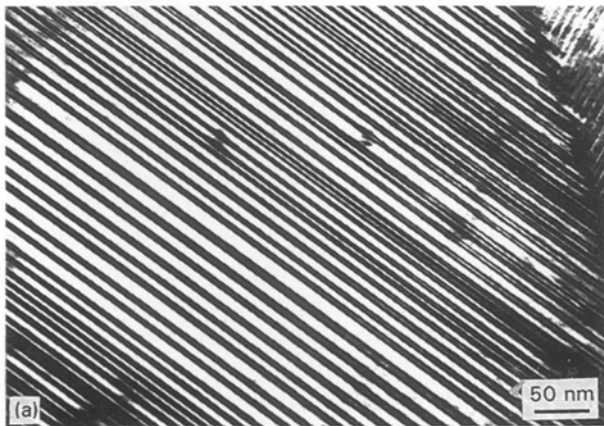


Figure 3 Intradendritic L1₀ martensite in Ni-24 at % Al-21 at % Cr aged for 140 h at 850 °C. (a) Bright-field micrograph; (b) selected-area diffraction pattern (**B** = [110]_{L1₀}).

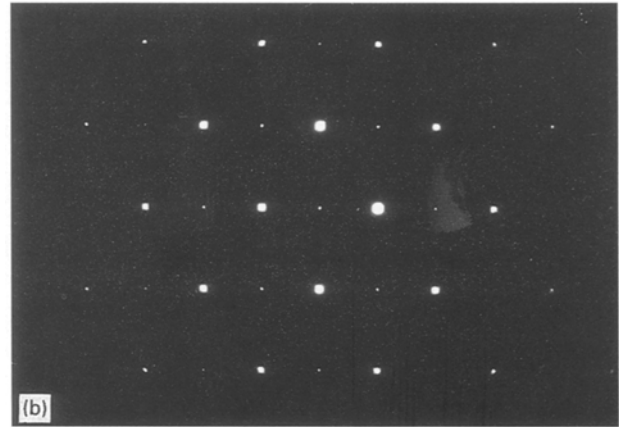
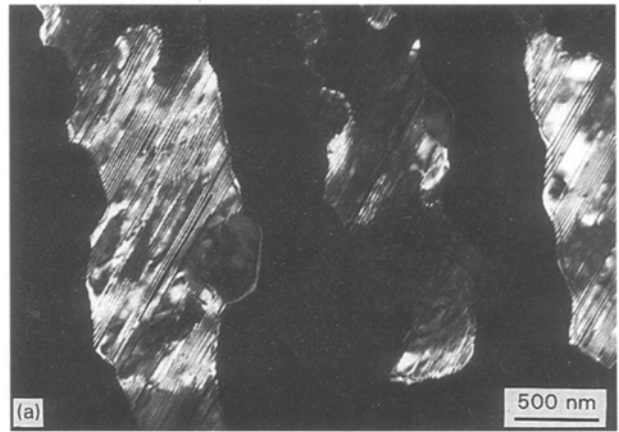


Figure 4 Intradendritic precipitation of γ' during ageing. (a) Twinned γ' formed during *in situ* ageing of Ni-24 at % Al-8 at % Cr-12 at % Co (dark-field image taken using **g** = (002)_{γ'} at a temperature of approximately 950 °C); (b) Selected-area diffraction pattern (**B** = [110]_{γ'}) identifying γ' formed in Ni-24 at % Al-21 at % Cr aged at 850 °C for 140 h.

such that

$$[110]_{L_{12}} \parallel [111]_{B_2}$$

$$(1\bar{1}1)_{L_{12}} \parallel (1\bar{1}0)_{B_2}$$

In general, the γ' precipitates were found to be twinned. Twinning of the γ' precipitates usually consisted of the formation of a central twinned midrib of the type described by Yang *et al.* [14] for γ' precipitated in a β-derived matrix.

Examination of the interdendritic regions revealed that ageing of the Ni-24 at % Al-15 at % Cr alloy for 140 h at 850 °C was found completely to remove the interdendritic γ-phase. Loss of the interdendritic γ-phase was found to occur as a direct result of growth of γ' at the expense of γ. No evidence was found for the interdendritic precipitation of α-Cr or other chromium-rich second phases during ageing of the Ni-24 at % Al-15 at % Cr alloy (although existing α-Cr remained stable during ageing). This contrasts with observations by Yang *et al.* [8] of an aged Ni-18 at % Al-9 at % Ti-10 at % Cr alloy for which loss of the γ-phase occurred as a result of removal of chromium from solid-solution in the interdendritic matrix by α-Cr precipitation during ageing.

In comparison with the Ni-24 at % Al-15 at % Cr material, both the intra- and interdendritic micro-

structures of the Ni–24 at % Al–21 at % Cr alloy were found to be relatively insensitive to ageing at 850 °C for 140 h. Following 850 °C/140 h ageing, the dendrite interiors of the 21 at % Cr alloy were found (Fig. 3) to consist almost entirely of L1₀ martensite (as in the as-cast condition). Isolated regions of the dendrite interiors which had transformed to a β-phase matrix containing γ' precipitates were observed. However, these β + γ' regions were confined to locations near the centres of the dendrites which were relatively aluminium-rich and chromium-lean compared to the bulk of the dendrites. Furthermore, following 850 °C/140 h ageing, complete transformation of the L1₀ martensite was observed in the case of the 15 at % Cr material, but not with the 21 at % Cr alloy.

Such intradendritic γ' precipitates (Fig. 4) as were observed in the 850 °C aged 21 at % Cr alloy were similar (in terms of size, morphology and orientation relationship to the β-phase) to those seen in the 15 at % Cr material. The following orientation relationship was observed in the 21 at % Cr alloy between the intradendritic β and L1₀ martensitic phases

$$[110]_{L1_0} \parallel [001]_{B2}$$

$$(1\bar{1}1)_{L1_0} \parallel (1\bar{1}0)_{B2}$$

The L1₀ martensite was found to be extensively twinned, as is commonly observed in the Ni–Al binary system.

The interdendritic γ/γ' microstructure of the as-cast Ni–24 at % Al–21 at % Cr alloy was largely retained in the 850 °C/140 h aged condition. Following 850 °C ageing, the interdendritic region was primarily composed of cuboidal γ' (around 500 nm on-edge) in a γ-matrix. These cuboidal γ' precipitates closely resembled those observed in the as-cast 21 % Cr alloy. Following 850 °C/140 h ageing, the formation of additional spheroidal γ' precipitates (typically with diameters of around 10–30 nm) was observed. A single-phase γ' layer with a thickness of around 1 μm was present around the dendrites in the as-cast condition. This layer was cube–cube orientation related to the interdendritic γ/γ' and did not have a detectable orientation relationship to the intradendritic L1₀ martensite. Following 850 °C ageing, the single-phase γ' layer was observed to have grown to a thickness of around 2–5 μm. Growth of the single-phase γ' layer occurred predominantly by advance into the dendrite interiors. However, a limited tendency for growth of the single-phase layer at the expense of the interdendritic-phase was observed. As in the case of the 15 at % Cr alloy, fresh interdendritic (or intradendritic) precipitation of α-Cr was not observed, although existing α-Cr precipitates remained stable.

In the case of the Ni–24 at % Al–21 at % Cr material, the interdendritic regions were strongly chromium enriched after 850 °C ageing when compared to those of the Ni–24 at % Al–15 at % Cr material. The presence of additional γ-stabilizing chromium correlated with the maintenance of a γ/γ' microstructure in the 21 at % Cr alloy and not in the 15 at % Cr material. Although the formation of new or enlarged α-Cr pre-

cipitates was not observed after ageing of either of the Ni–Al–Cr alloys investigated, extensive α-Cr precipitation was noted in the as-cast condition. By removing chromium from solution in the interdendritic matrix, this α-Cr precipitation presumably contributed to increasing the overall level of chromium required to maintain the interdendritic γ-phase during ageing.

The changes induced, by ageing at 850 °C for 140 h, in the intradendritic microstructure of the Ni–24 at % Al–8 at % Cr–12 at % Co alloy were found generally to resemble those in the Ni–24 at % Al–15 at % Cr material. As with the 15 at % Cr alloy, the 140 h at 850 °C ageing treatment brought about complete transformation of the L1₀ martensite. Following ageing, the cobalt-bearing alloy was found to have an intradendritic microstructure composed of a β-phase matrix containing γ' precipitates. The precipitation of γ' in the cobalt-containing alloy (Fig. 4) occurred with the following orientation relationship to the β-phase

$$[110]_{L1_2} \parallel [001]_{B2}$$

$$(1\bar{1}1)_{L1_2} \parallel (1\bar{1}0)_{B2}$$

The γ' phase formed as internally twinned plates with a morphology somewhat similar to that of the L1₀ martensite in the as-cast cobalt-bearing alloy. Despite the morphological similarity of the martensite and the γ' plates, hot-stage TEM observations suggest that the γ' did not form *in situ* from the martensite. Instead, the γ' precipitates were nucleated directly from the β-phase, immediately after the transformation of the dendrite matrix from L1₀ to B2.

As in the case of the Ni–24 at % Al–15 at % Cr material, ageing of the Ni–24 at % Al–8 at % Cr–12 at % Co alloy for 140 h at 850 °C was found to result in complete removal of the interdendritic γ-phase. However, the circumstances under which loss of the interdendritic γ-phase occurred differed between these two alloys. As was noted above, fresh precipitation of α-Cr was not observed during ageing of the Ni–24 at % Al–15 at % Cr material. Hence, in the cobalt-free alloy, loss of γ-stabilizing chromium (from solid-solution in the interdendritic matrix), during ageing, did not contribute to destabilization of the interdendritic γ-phase. In contrast, in the cobalt-bearing alloy, extensive fresh precipitation of α-Cr was observed. Unlike the α-Cr formed in the as-cast condition, these α-Cr deposits were rich in cobalt when compared with the interdendritic matrix, and hence served as a sink for γ-stabilizing cobalt and chromium. Some of the larger (2–3 μm or greater in length) α-Cr precipitates appeared to be two-phase near their cores. However, differential thinning problems prevented identification of the second phase.

In Yang *et al.*'s study [8] of an Ni–18 at % Al–9 at % Ti–10 at % Cr alloy, α-Cr precipitation was observed to occur *in situ* within the chromium-rich γ-phase. In the present investigation, the α-Cr precipitated in the Ni–24 at % Al–8 at % Cr–12 at % Co alloy was typically in the form of finger or hand-like

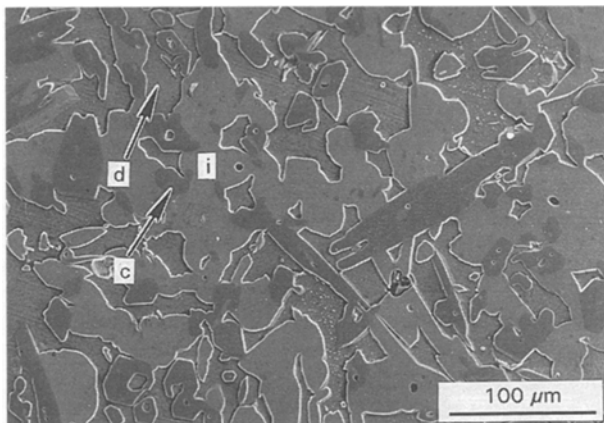


Figure 5 Secondary electron image of Ni-24 at % Al-21 at % Cr aged for 140 h at 850 °C (d = L1₀ type martensitic dendrite interior, i = interdendritic γ/γ' , c = α -Cr).

deposits with lengths of around 1–5 μm and widths of around 500 nm. These α -Cr deposits were apparently evenly distributed throughout the interdendritic region. The morphology and distribution of the α -Cr precipitates present after ageing do not readily correlate with those of the interdendritic γ -phase observed in the as-cast condition. Hence, in the present investigation, precipitation of α -Cr cannot have occurred *in situ* within the γ -phase. Nonetheless, the removal of both chromium and cobalt from the interdendritic matrix by α -Cr precipitation correlates with loss of the γ -phase from the interdendritic regions during ageing.

The Ni-24 at % Al-21 at % Cr alloy was selected for further investigation (Fig. 5), because this was the only material which retained the interdendritic γ/γ' microstructure after 850 °C ageing. The tendency for growth of the single-phase γ' layer (present in the as-cast condition around the dendrites) was somewhat more marked after 1100 °C ageing than in the case of 850 °C treatment. Typically, after 140 h ageing at 1100 °C, the single-phase γ' layer had grown from around 1 μm in thickness (as-cast) to around 5–7 μm . In comparison, after 850 °C ageing, the single-phase γ' layer was around 2–5 μm thick. In all other respects, ageing of the Ni-24 at % Al-21 at % Cr alloy for 140 h at 1100 °C was found to yield microstructures closely resembling those observed after 850 °C ageing.

In all three of the Ni-Al-Cr-(Co) alloys examined in the present work, no evidence was found of the formation of Ni₅Al₃. However, given that the lowest ageing temperature employed was 850 °C, the absence of the (low-temperature) Ni₅Al₃ phase is not surprising. It is, however, entirely possible that Ni₅Al₃ would have been precipitated if a lower ageing temperature had been employed. For example, in work on binary Ni-Al alloys [15], Ni₅Al₃ formation is observed after ageing at 400 °C.

In summary, of the materials examined, only the Ni-24 at % Al-21 at % Cr alloy shows promise for the production of stable interdendritic γ/γ' . Furthermore, the formation of interdendritic γ/γ' is achieved at the price of the formation of intradendritic L1₀ martensite (rather than β -phase). Clearly, given that β - γ/γ' is produced on ageing of the coating analogue

alloy examined in previous work [10], further work is required. Based on initial work by the authors on a range of Ni-Al-Cr alloys containing 27 at % Al, an increase in the aluminium content merely serves to form single-phase interdendritic γ' in the as-cast condition, rather than γ/γ' , whilst the dendrite interiors remain martensitic.

4. Conclusions

An investigation has been conducted into the microstructural stability, during ageing, of cast Ni-24 at % Al-21 at % Cr, Ni-24 at % Al-15 at % Cr and Ni-24 at % Al-8 at % Cr-12 at % Co alloys. In the as-cast condition, the materials investigated consisted of dendrites with an L1₀ type martensitic matrix plus interdendritic γ/γ' . The γ/γ' microstructure was retained in a largely unchanged condition in an Ni-24 at % Al-21 at % Cr alloy aged at either 850 or 1100 °C for 140 h. In contrast, complete transformation of the interdendritic γ -phase to γ' occurred in a Ni-24 at % Al-15 at % Cr alloy aged for 140 h at 850 °C.

No evidence was found in either the 21 at % Cr or the 15 at % Cr alloy for removal of γ -stabilizing chromium from the interdendritic matrix by the precipitation of α -Cr during ageing. However, in both the 21 at % Cr and the 15 at % Cr materials, α -Cr formed during casting remained stable during ageing and this precipitation served to trap chromium during ageing. In the Ni-24 at % Al-8 at % Cr-12 at % Co alloy, extensive precipitation of α -Cr during ageing at 850 °C for 140 h removed γ -stabilizing chromium and cobalt from the interdendritic matrix. In the cobalt-bearing alloy, the interdendritic matrix was transformed to single-phase γ' after 140 h ageing at 850 °C.

In both the 15 at % Cr alloy and the cobalt-bearing material, transformation of the intradendritic L1₀ martensite occurred during ageing at 850 °C for 140 h. Following ageing, these two materials exhibited dendrites composed of a β -phase matrix plus γ' precipitates. In contrast, L1₀ martensite was observed within the dendrites of the Ni-24 at % Al-21 at % Cr alloy after ageing at either 850 or 1100 °C for 140 h.

Acknowledgements

The research described in this paper was partially sponsored by the Division of Materials Sciences, US Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc. and through the SHaRE Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities. W.F.G. wishes to thank Dr Neal Evans, Oak Ridge National Laboratory, for his co-operation during the use of SHaRE facilities.

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*Received 10 August
and accepted 8 September 1995*