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Precipitation in Ni–Co–Al alloys

Part 2 *Discontinuous precipitation*

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Discontinuous precipitation of the γ' phase in Ni–Co–Al alloys has been studied in the temperature range 673 to 973 K using optical microscopy and transmission electron microscopy. Discontinuous precipitation was observed to occur in all the alloys to some degree, the extent of the precipitation decreasing with decreasing cobalt content of the alloy. Only in high cobalt alloys (greater than 38 at %) did the discontinuous transformation go to completion. The discontinuous transformation product consisted of fine straight coherent γ' lamellae with a specific cube–cube orientation relationship to the γ matrix at low ageing temperatures and short times. At higher temperatures and longer times, continuous and discontinuous coarsening processes produced a coarse, less regular structure with a plate spacing ten times that of the regular structure. The overall kinetics of the discontinuous transformation were affected by the prior continuous precipitation of γ' particles ahead of the discontinuous reaction fronts. The coarsening of these continuous particles as a function of time stopped the transformation in low cobalt alloys and continuously slowed the rate of transformation in high cobalt alloys resulting in Avrami exponents, n, less than 1. Reversion experiments and experiments on prestrained specimens yielded Avrami exponents of $n = 1$ lending support to the above suggestion. The major effect of cobalt in these alloys was to increase their propensity to transform discontinuously. This effect was largely due to the decrease in the rate of continuous precipitation and coarsening as a result of the increased partitioning of cobalt between γ and γ' in high cobalt alloys.

1. Introduction

Decomposition of nickel based alloy solid solutions precipitating a γ' phase can occur either continuously or discontinuously. The continuous process occurs throughout the grains and results in a microstructure consisting of cubes or spheres of the face centred cubic ordered phase γ' in a face centred cubic γ matrix (Fig. 1a). The discontinuous process usually originates at grain boundaries resulting in plates of γ' in a γ matrix (Fig. 1b). The processes are competitive and continuous precipitation frequently confines discontinuous precipitation to narrow grain boundary regions (Fig. 2), as is the case in many commercial nickel base alloys, given conventional heat treatments [1–5]. However, extensive discontinuous precipitation has been observed in a number of relatively simple nickel base alloys [2, 6–16]. Published data suggest that addition of cobalt to nickel based alloys promotes the discontinuous transformation [2, 7–9]. The present paper is the sequel to that previously published on continuous precipitation in Ni–Co–Al alloys [17] and reports work on the effect of cobalt addition on the extent and nature of the discontinuous transformation.

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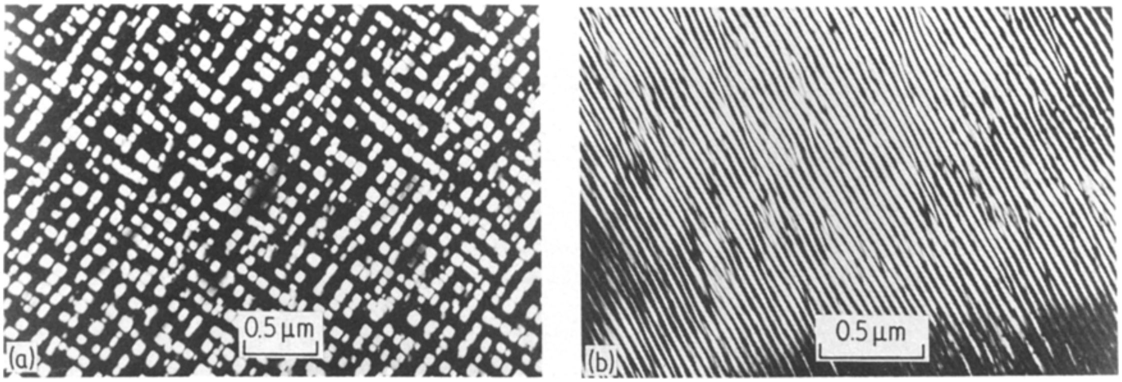

Figure 1 Dark field electron micrographs of Ni 48–9 imaged using a strong 0 1 0 γ' superlattice spot. (a) Aged at 1033 K for 24 h showing continuous precipitation of aligned cube shaped γ' particles. (b) Aged at 873 K for 7 h showing discontinuous precipitation of plates of γ'.

2. Experimental procedure

Nine Ni–Co–Al alloys were prepared by vacuum melting and casting 99.99% purity starting materials. The results of chemical analysis are shown in Table I together with the preparation methods used to produce material in the form of 7 mm diameter rods. The alloys are designated by a code giving their nominal compositions in at %. Thus Ni 48–9 contains 48 at % Co and 9 at % Al.

Solution and ageing treatments were carried out in a vertical tube vacuum furnace under a vacuum of 10^{-6} torr followed by a water quench.

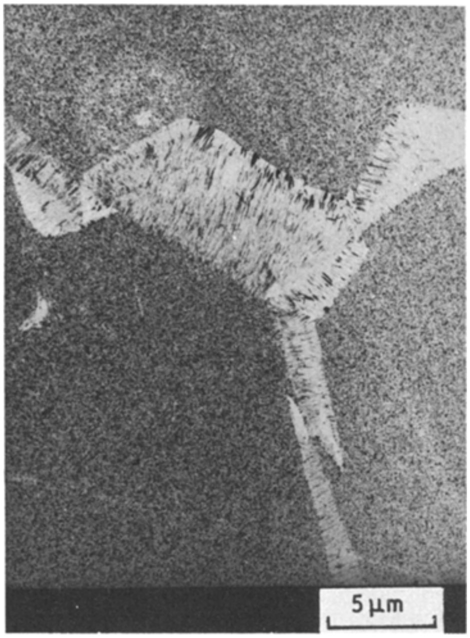

Figure 2 Electron micrograph of extraction replica of Ni 37–12 aged for 19 h at 1023 K showing a grain boundary zone of discontinuous precipitate.

Solution treatment was carried out at the temperatures shown in Table I for 1 h and ageing treatments of 673 to 1023 K for various times. Specimens for optical and scanning electron microscopy were machined from the rods, mechanically ground and polished to a 1 μm diamond finish.

For optical microscopy, solution treated specimens were electrolytically etched in a solution of 5% HF + 10% glycerol in water and aged specimens were etched in 2% ammonium sulphate + 2% citric acid in water. The latter etchant was also used in the preparation of carbon extraction replicas and in the preparation of specimens for scanning electron microscopy. Thin foil specimens were cut from 3 mm diameter rod and ground until approximately 0.25 mm thick with parallel faces. Thinning was carried out in a two stage jet polishing apparatus with an electrolyte of 10% perchloric acid in ethanol.

Transmission electron microscopy was carried out on either a Jeol JXA-7A or a Jem 100-CX electron microscope, extraction replicas being examined at 80 kV and thin films at 100 kV. Scanning electron microscopy was carried out on a Jeol JXA-50A microscope.

3. Results and discussion

3.1. Occurrence of discontinuous transformation

Large discontinuously transformed regions were only found in alloys which had been transformed in the temperature range 673 to 973 K. At higher temperatures continuous precipitation was the dominant mode of transformation [17].

TABLE I Alloy compositions

Alloy name	Alloy composition (at %)			Solution treatment temperature (K)	Grain size (μm)	Preparation
	Ni	Co	Al			
Ni 0–13	86.7	–	13.3	1293	180	Homogenized 24 h
Ni 10–13	77.9	9.5	12.6	1293	150	at 1473 K,
Ni 22–13	64.9	21.7	13.4	1343	145	forged and
Ni 37–12	50.4	37.1	12.5	1293	140	swaged at 1373 K
Ni 49–12	39.3	48.7	12.0	1293	100	
Ni 9–12	78.9	9.3	11.79	1223	80	Extruded at 1323 K
Ni 22–11	67.1	22.1	10.77	1223	34	Homogenized 17 h
Ni 38–8	53.4	38.2	8.38	1223	40	at 1373 K
Ni 48–9	43.3	47.8	8.97	1223	67	swaged at 1373 K

Even within this temperature range the processes are competitive, with prior continuous precipitate stopping or slowing the discontinuous transformation.

The Ni–Al alloy only ever exhibited very narrow grain boundary regions which had transformed discontinuously. Alloys Ni 22–11 and Ni 22–13 exhibited regions consisting of up to 25% of the specimen which had transformed discontinuously at some temperatures. The rates of transformation were always slow and at no temperature did the transformation go to completion. In all four high cobalt alloys the specimens completely transformed discontinuously at some temperature. The extent of the discontinuous transformation clearly increased as the cobalt content of the alloys increased as was anticipated from published data [2, 7–9].

The times necessary to cause transformation at a given temperature are shown in the TTT diagrams (Fig. 3). The fractions transformed were measured by optical microscopy. These diagrams show the times taken to achieve both 0.05 and 0.95 volume fractions transformed at a given temperature in the high cobalt alloys. The times were normalized by dividing by the grain boundary area per unit volume (A) in order to remove the effect of the different grain sizes. A comparison of Fig. 3b with 3a and Fig. 3d with 3c shows that the major effect of decreased aluminium content is to move the curves to lower temperatures. This presumably results from the decrease in solvus temperature with decreasing aluminium content and the corresponding decrease in supersaturation and driving force. Comparison of Fig. 3c with 3a and Fig. 3d with 3b shows that the major effect of the decreasing cobalt content is to move the curves to longer times. This evidence, together with that referred to above, clearly suggests that the effect of cobalt in the alloys is to increase the propensity for the alloy to transform discontinuously.

3.2. The precipitation process

The majority of studies were carried out on high cobalt alloys where it was possible for the discontinuous reaction to completely transform the specimen. Studies were also carried out on other alloys at temperatures where significant amounts of discontinuous transformation occurred, and particularly to study the effect of prior continuous precipitation on the discontinuous transformation.

3.2.1. Initiation of the discontinuous transformation

The transformation was initiated by γ' precipitates forming preferentially on one side of a grain boundary (Fig. 4a). These γ' nuclei had a typical separation of 0.25 μm. The grain boundary in these regions then migrated into an adjoining grain becoming a reaction front for the discontinuous transformation (Fig. 4b). Several reaction fronts formed on a given grain boundary, some causing the boundary to migrate in opposite directions in an "S" configuration. No precipitation occurred on twin boundaries. The γ' precipitate lamellae appear to extend beyond the reaction front. This suggests that the reaction front is pulled along by the growing γ' particles rather than the precipitation process occurring behind an already migrating boundary [18–20]. This observation closely resembles that reported by Ness and Billdal [21] for Al–Zr alloys.

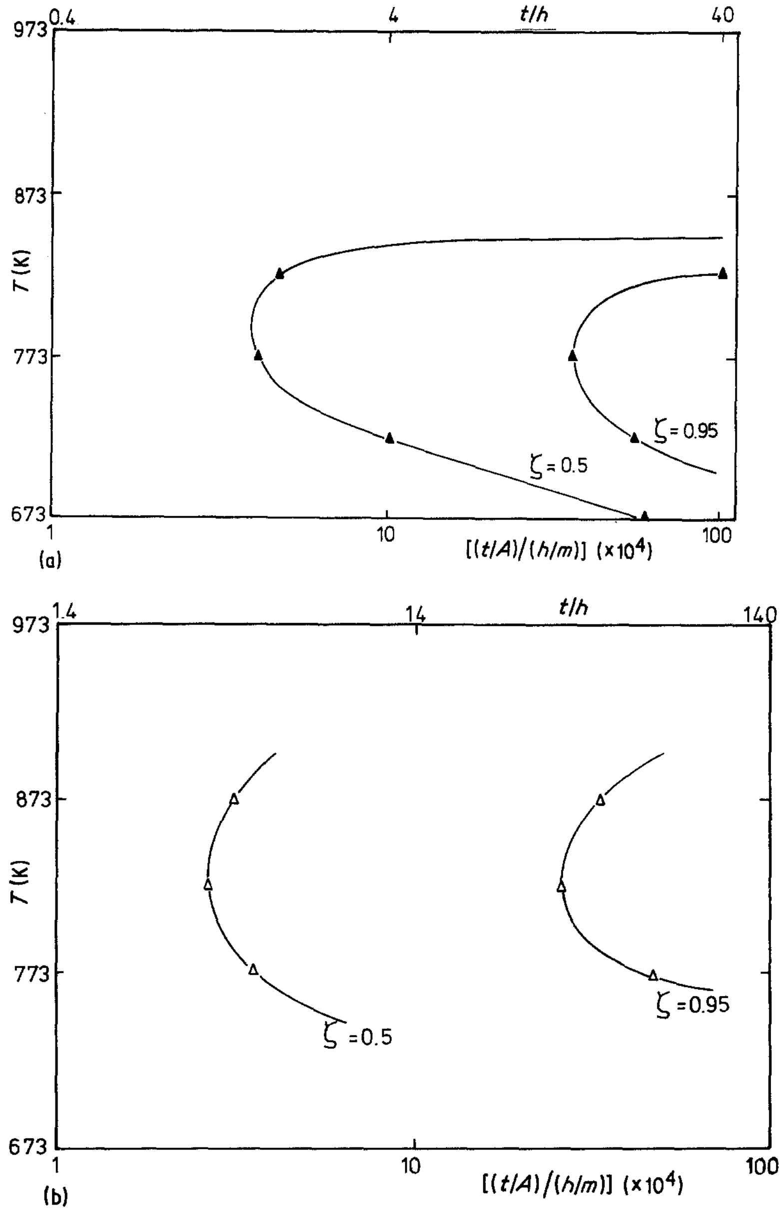

Figure 3 TTT Diagrams. Note that the time, t, has been divided by the area, A, of grain boundary per unit volume to compensate for different grain sizes. (a) Ni 38–8. (b) Ni 37–12.

3.2.2. Growth of the discontinuous precipitate

At temperatures where the driving force for discontinuous precipitation is high, the initial growth morphology of the discontinuous precipitate consists of parallel lamellae of γ and γ' with a constant interlamellar spacing behind a planar growth front (Figs. 5a and b). Selected area diffraction studies have shown that the orientation of a discontinuous region is the same as that of the grain from which it is growing. Further evidence of this expected orientation relationship is given by the fact that the twin boundaries in the grain from which the discontinuous region is growing are reproduced in the discontinuously transformed structure.

During discontinuous precipitation where the products are precipitated simultaneously, a specific crystallographic orientation relationship between the precipitate and the transformed matrix is often observed [6, 9, 22–26]. Fig. 1b is a centred dark field electron micrograph of the discontinuous precipitate imaged with a strong 0 1 0 γ' superlattice spot. The micrograph shows that the discontinuous precipitates are all clearly resolved and are aligned parallel to

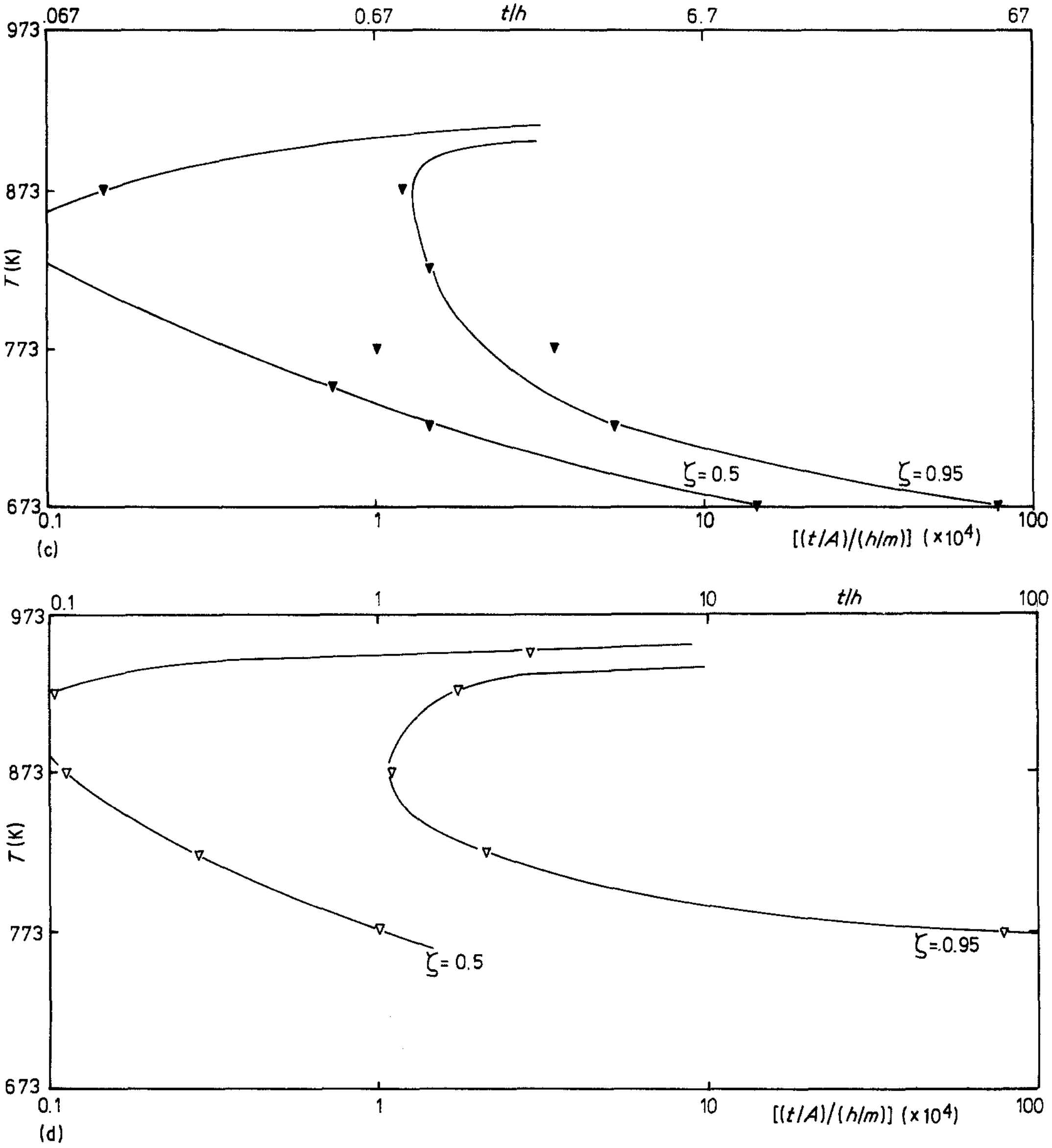

Figure 3 Continued (c) Ni 48–9. (d) Ni 49–12.

the $\langle 1 0 0 \rangle$ directions in the γ matrix. This experiment was repeated in many regions and the crystallographic orientation relationship between the γ' precipitate lamellae and the depleted γ matrix phase could always be described by

$$[0 0 1]_\gamma \parallel [0 0 1]_{\gamma'}; \qquad (0 0 1)_\gamma \parallel (0 0 1)_{\gamma'}$$

This orientation relationship is the same as that observed during continuous precipitation of γ' in Ni–Co–Al alloys at higher ageing temperatures [10–12, 17] and in other γ' precipitation hardened alloys [27, 28]. The lattice parameter mismatch between γ and γ' when precipitated in a continuous manner in the high cobalt alloys at high temperatures is approximately 0.3% [17] and the γ' particles are coherent with the matrix. The fact that the orientation relationship for the two modes of precipitation is similar and that the γ' plates are long, straight and parallel suggests that the discontinuous γ' precipitate is coherent or semicoherent with the γ matrix, at least in the early stages of precipitation. This suggestion is further supported by the fact that no interfacial dislocations have been observed at the precipitate–matrix interface.

As the volume fraction of the discontinuous

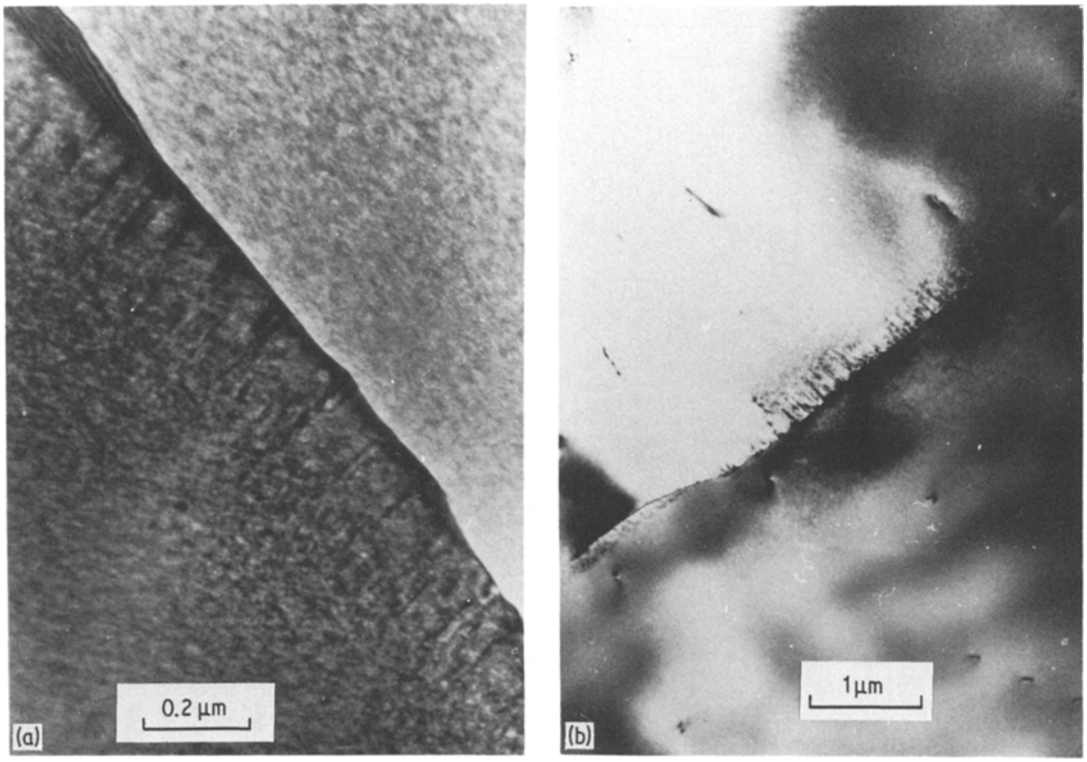

Figure 4 Ni 48–9 aged for 1 h at 873 K (a) Initial γ' precipitation at a grain boundary. (b) Reaction fronts on a grain boundary moving in opposite directions.

precipitate increases with ageing time, sidewise growth of colonies occurs and the lamellae multiply in order to maintain a reasonably constant lamellar spacing. Lamellation can be accomplished by lamellae branching [18, 21, 26, 29] or by repeated nucleation of new lamellae at the advancing interface [30, 31]. A combination of these mechanisms seems to occur in Ni–Co–Al alloys (Figs. 6a and b) with lamellar branching being by far the most prominent.

In Ni 48–9 the streamline flow pattern of the approximately parallel γ' lamellae is interrupted by regular structural discontinuities (Figs. 7a and b). These discontinuities sometimes extend over several hundreds of micrometres and numerous colonies were dissected by up to three discontinuities running sidewise across them. At a discontinuity there appears to be a local change of lamellar spacing although this is constant either side of the discontinuity (Fig. 7b). A

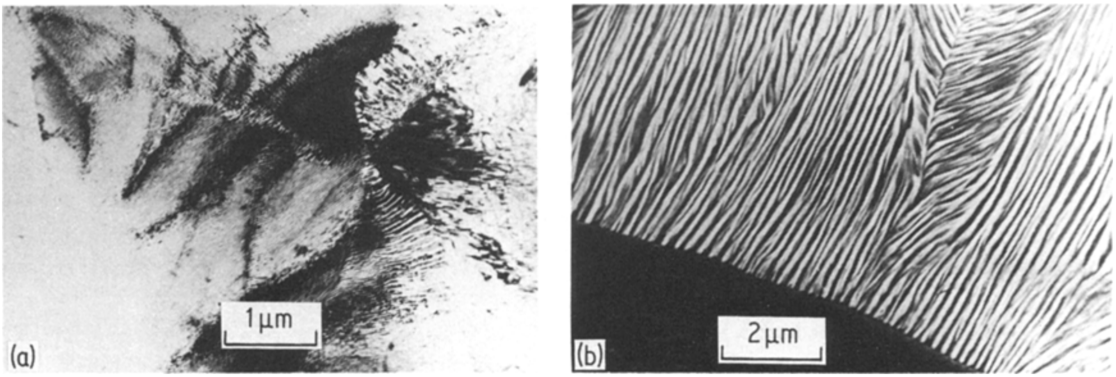

Figure 5 (a) Transmission electron micrograph of Ni 37–12 aged for 0.5 h at 873 K. (b) Scanning electron micrograph of Ni 48–9 aged for 2 h at 873 K.

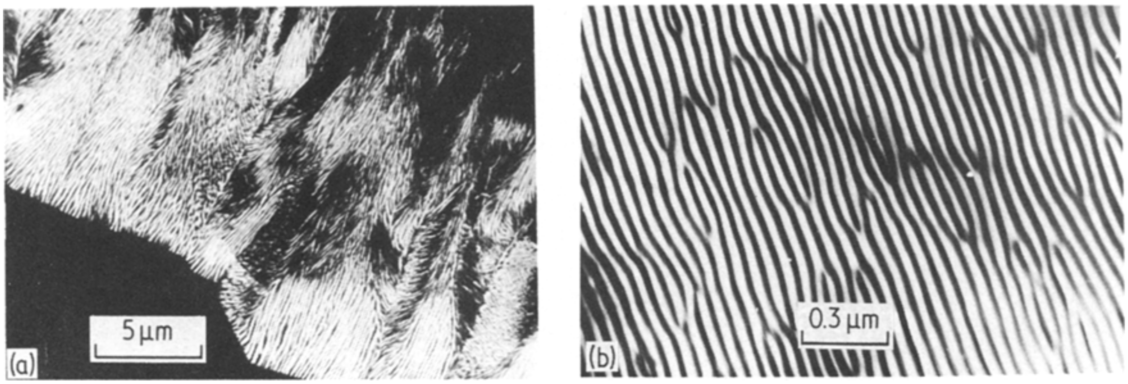

Figure 6 (a) Scanning electron micrograph of Ni 48–9 aged for 2 h at 873 K. (b) Transmission electron micrograph of Ni 48–9 aged for 7 h at 873 K. The micrographs show lamellation accomplished largely by lamellar branching.

similar structural feature has been observed during discontinuous transformation in Al–Zr alloys [21]. The discontinuities were attributed to plate growth following selected low-energy coincidence site lattice orientations.

At longer ageing times, particularly at higher temperatures, the γ' lamellae within colonies becomes less regular. A considerable variation in the plate orientation and plate thickness has been observed from colony to colony. These effects may be largely due to the effect of prior continuous precipitation and simultaneous or subsequent coarsening of the discontinuous transformation product.

3.2.3. Coarsening of the discontinuous precipitate

Discontinuous coarsening occurred at impinged reaction fronts or cell boundaries, and at the sites of the original grain boundaries. The reaction

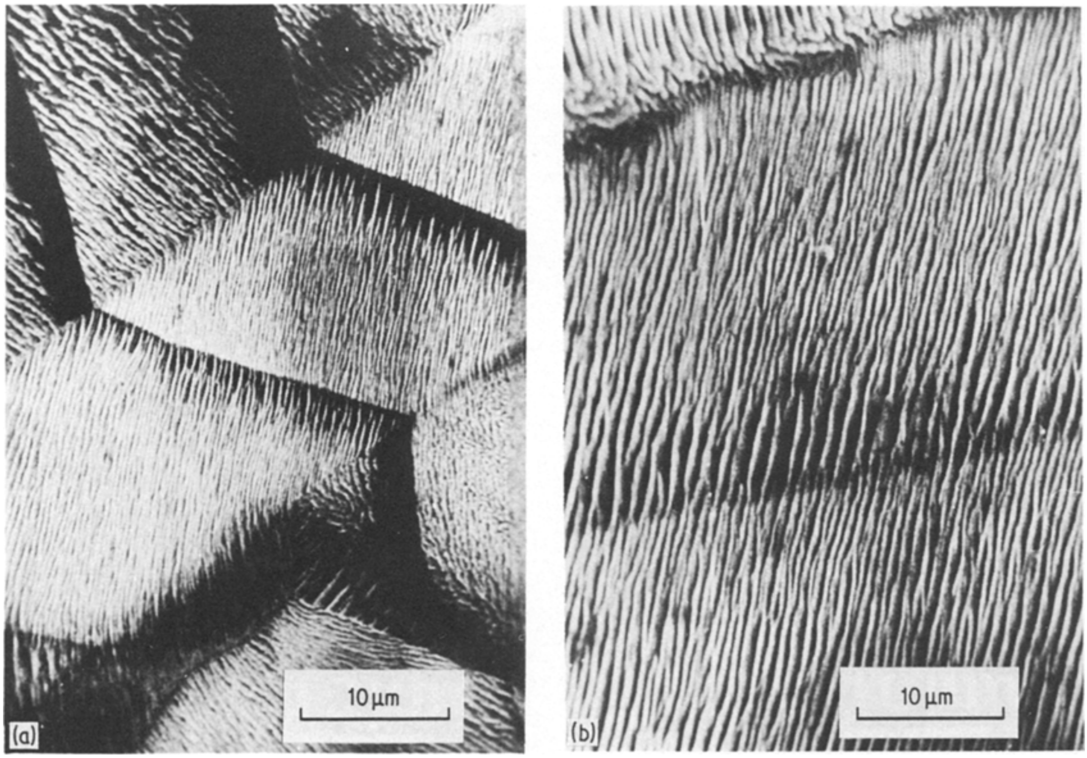

Figure 7 (a) and (b) Scanning electron micrographs of Ni 48–9 aged for 5 h at 873 K showing regular structural discontinuities.

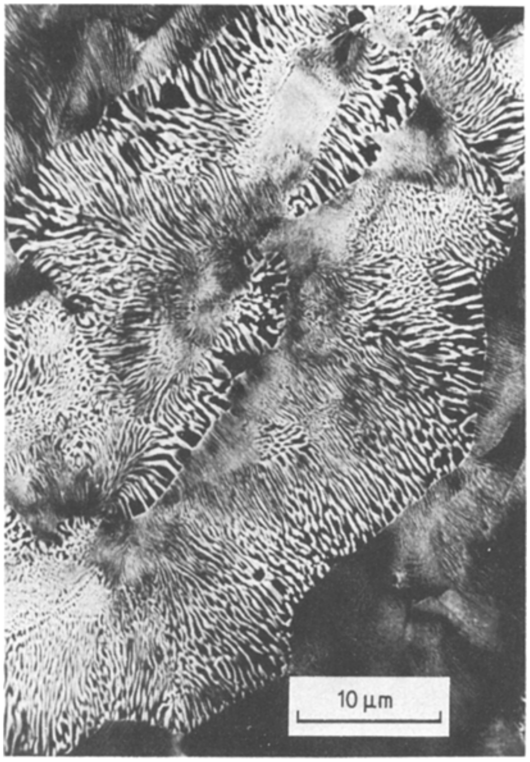

Figure 8 Scanning electron micrograph showing secondary discontinuous reaction fronts in Ni 48–9 aged for 7 h at 873 K.

occurred by boundary migration with the boundary migrating into regions whose lamellae were most nearly normal to the boundary plane (Fig. 8). As the coarsening reaction front swept across a colony the fine γ' precipitate lamellae were transformed into irregular, coarse γ' lamellae with an increased interlamellar spacing of the order of 10 times. In alloys in which the extent of the initial discontinuous transformation was limited the discontinuous coarsening reaction consumed most of the already transformed material. In high cobalt alloys which were completely transformed by the initial discontinuous transformation the discontinuous coarsening reaction consumed up to 25% of the already transformed material, the secondary reaction commencing prior to complete transformation of the specimen by the primary reaction but proceeding at a much slower rate.

Discontinuous coarsening has been observed in a number of alloy systems [13, 32–37]. The growth configuration is dependent on whether the growth fronts at a boundary migrate from one or both sides of the boundary. Growth in two alternating directions results in a typical "S" configuration [33] which at a later stage of

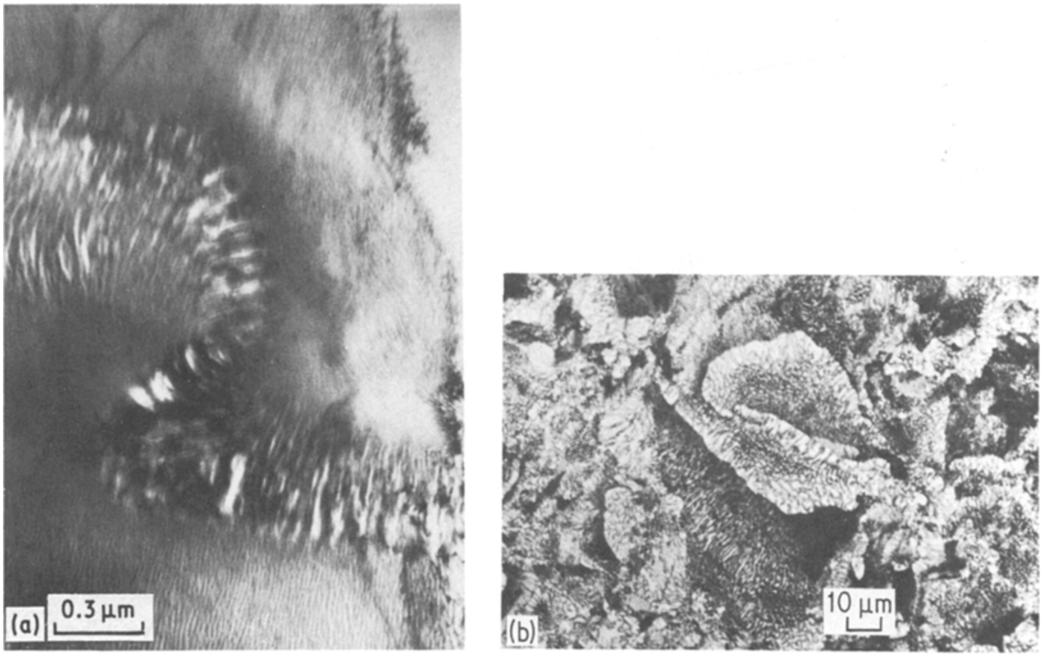

Figure 9 (a) Transmission electron micrograph of Ni 37–12 aged for 0.5 h at 873 K showing an "S" shaped secondary discontinuous reaction front. (b) Optical micrograph of Ni 48–9 aged for 200 h at 873 K showing "S" shaped secondary discontinuous reaction fronts.

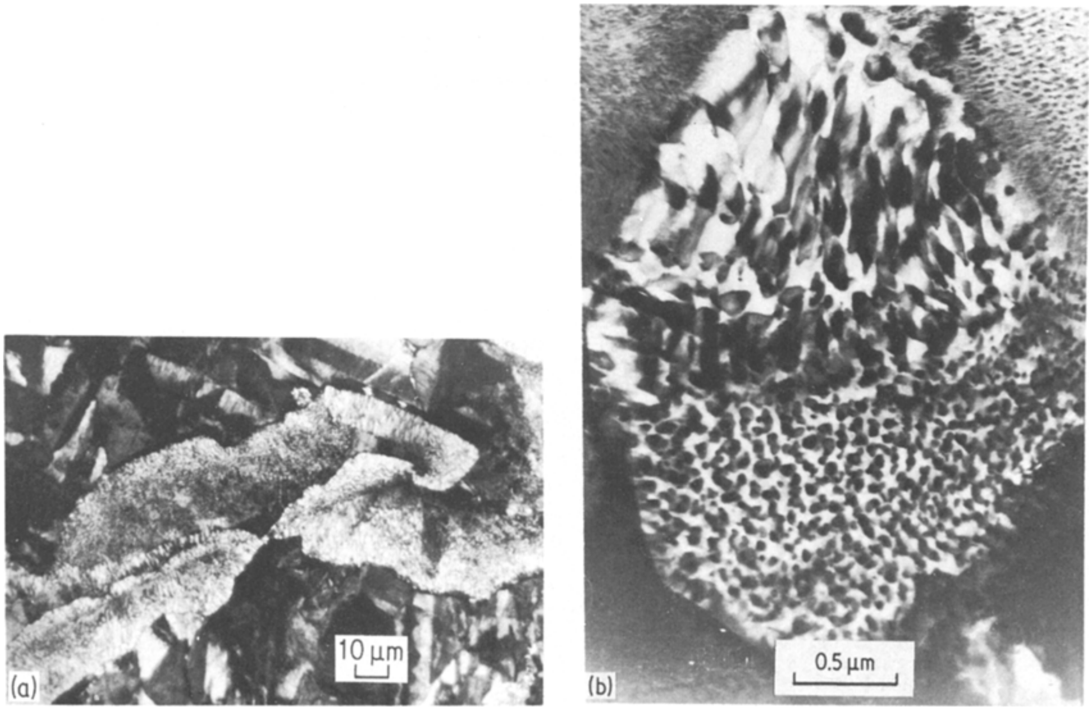

Figure 10 Optical micrograph (a) and transmission electron micrograph (b) of Ni 48–9 aged for 24 h at 873 K. (a) Shows the development of a double seam structure via "S" shaped secondary discontinuous reaction fronts. (b) Shows a double seam structure which consists of two hemispherical secondary discontinuous colonies which abut along a boundary.

growth develops into a double seam of discontinuously transformed material, formed on either side of the boundary [37]. Growth on a single side of the boundary usually results in one single seam on one side of the boundary only. In Ni–Co–Al alloys the "S" mechanism is the predominant transformation configuration for both the primary discontinuous precipitation reaction and particularly for the secondary discontinuous coarsening process. Examples of nucleation on alternate sides of a boundary producing an "S" structure can be seen in Figs. 9a and b. Figs. 10a and b show examples of the development of an "S" structure into a double seam type configuration. The "S" morphology of discontinuous transformation has been reported to predominate at lower temperatures [19, 37, 38] where the driving force for the discontinuous transformation is high, resulting in a high initial nucleation density. This is in accord with the situation in Ni–Co–Al alloys where the discontinuous transformation is only observed at temperatures below that at which the continuous transformation predominates.

The primary discontinuous transformation product also coarsens continuously at long ageing times. The continuous reaction is first observed at lamella facets or terminations within cells. The reaction took place by shrinkage of the terminations, resulting in a noticeable increase in thickness of the adjacent lamellae. This preferential coarsening of the thick lamellae at the expense of the shrinking smaller lamellae gradually reduces the number of lamellae, producing a coarser less regular structure.

The discontinuous and continuous coarsening reactions are in a sense competitive. Both reduce the energy of the system by reducing the γ' plate surface area per unit volume and both drive the γ matrix composition towards its equilibrium value. The driving force for discontinuous coarsening is progressively reduced as the primary discontinuous transformation product coarsens continuously. The driving force is eventually reduced to such a value that discontinuous coarsening ceases. This point always seems to occur in the present alloys before 30% of the primary discontinuous reaction product has coarsened discontinuously. The extent of discontinuous coarsening is greater at lower temperatures, which is to be expected as the process takes place by grain boundary diffusion whereas continuous coarsening occurs via volume diffusion.

3.3. Overall kinetics of discontinuous transformation

The volume fraction of specimens transformed by the discontinuous transformation was measured as a function of time at temperatures of 673 to 948 K, using optical microscopy. In general the volume fraction transformed as a function of time could be represented by a sigmoidal curve. It was not possible to make useful measurements on the lower cobalt alloys as the discontinuous transformation only transformed small fractions of the specimens. The results show that the fraction transformed discontinuously at a given time and temperature increases with increasing cobalt content of the alloys as has been previously illustrated in this paper by the use of TTT diagrams.

Following site saturation at the grain boundaries the overall kinetics of the discontinuous transformation would be expected to follow an Avrami type equation [39–41]:-

$$\zeta = 1 - \exp(-kt^n)$$

where ζ is the fraction transformed at a time t, k is constant at a given temperature, if the growth rate is constant, and $n = 1$, following site saturation.

TABLE II Constants from the Avrami Equation

Alloy	Temp (K)	n	k
Ni 38–8	673	0.71	−3.68
	723	0.85	−3.68
	748	0.74	−3.27
	773	0.68	−2.71
	823	0.46	−1.89
Ni 37–12	773	0.65	−2.94
	823	0.45	−1.95
	873	0.74	−3.28
	898	0.46	−1.91
Ni 48–9	673	0.79	−3.74
	723	1.13	−4.16
	748	0.53	−1.91
	773	0.99	−3.82
	823	0.35	−0.9
	848	0.89	−3.10
	873	0.64	−1.79
Ni 49–12	823	0.80	−2.71
	873	0.67	−1.92
	898	0.74	−2.41
	923	0.51	−1.54
	948	0.33	−1.52
Ni 22–11	673	0.71	−4.11
	748	0.63	−3.12
	823	0.18	−1.55

A plot of $\log \ln[1/(1 - \zeta)]$ against $\log t$ would be a straight line with a slope of 1 and the value of k would be the intercept on the $\log \ln[1/(1 - \zeta)]$ axis. The fractions transformed obtained from the optical microscope study were plotted in the above manner and the values of n and k obtained by a least-squares fit are shown in Table II. The values of the exponent n can be seen to be generally less than 1, although the value tends to increase towards 1 with decreasing temperature and with increasing cobalt content in the alloy. This result is not dissimilar to that obtained for a Co–Ni–Ti–Al alloy [7, 8] transformed discontinuously at temperatures between 723 and 923 K, when the exponent n was found to be approximately 0.7. In a Ni–Co–Ti alloy, it was found that the data on a $\log \ln[1/(1 - \zeta)]$ against $\log t$ plot could not be fitted by a straight line [7, 8]. It was concluded that time dependent continuous precipitation ahead of the discontinuous transformation front continually slowed the growth rate producing "apparent" values of n of less than 1. Continuous precipitation has often been observed to occur simultaneously with discontinuous precipitation in a large number of alloy systems [19, 20]. While in some systems the presence of continuous precipitate limits the extent of discontinuous precipitation, in others discontinuous precipitation proceeds to completion [6–8, 21, 23, 33, 42–44], often absorbing the prior continuous precipitate. In the present case the presence of continuous precipitate limits the extent of discontinuous precipitation in low cobalt alloys and prevents discontinuous precipitaton occurring in all alloys at elevated temperatures. Prior continuous precipitation is probably present in all alloys at all temperatures as only very rapid quenching prevents the continuous precipitate forming on cooling from the solution treatment temperature [17]. In most alloys the total volume fraction of continuous precipitate comes out of solution very early during the ageing process [17]. Even in the alloys where the discontinuous transformaton goes to completion the discontinuous precipitate can be seen to be consuming quite coarse continuous precipitate at the reaction front at long ageing times. Several authors have attempted to develop expressions for the discontinuous growth rate in situations where the discontinuous precipitate grows into prior continuous precipitate [42, 45–47]. This growth rate will become time dependent, due largely in the present case to the coarsening of continuous precipitate ahead of the discontinuous reaction front. The equation developed by Hornbogen [46] has been used to estimate the time dependence of the discontinuous growth rate in Ni–Co–Al alloys [11]. The time dependence of the solute concentration and γ' particle sizes ahead of the moving discontinuous transformation front were estimated using the Liftshitz, Slyosov Encounter Modified (LSEM) coarsening theory [48]. The procedure leads to an equation for the fraction transformed (ζ) which would yield "apparent" values of $n < 1$. The value of n would approach one at lower temperatures when the coarsening rate of continuously precipitated γ' particles becomes slow. Due to the increasing segregation of Co between γ and γ' with increasing Co content the coarsening rate of continuously precipitated γ' particles was found to decrease with increasing cobalt content [17]. This leads to estimates of n which are more nearly one in high cobalt alloys.

Specimens of Ni 38–8 which had been aged for 2 h at 750 K and exhibited 30% by volume of discontinuous precipitate were reheated to 1008 K. The discontinuous precipitate was dissolved by the reaction fronts sweeping back to the original grain boundaries. The fraction dissolved as a function of time was measured by optical microscopy. The data were plotted as $\log \ln[1/(1-\zeta)]$ against $\log t$ and yielded values of n near 1 (Fig. 11). This supports the previous conclusion that the values of $n < 1$ were due to continuous precipitation ahead of the advancing boundary.

Specimens of Ni 38–8 were prestrained in an Instron tensile machine prior to ageing at 750 K. The fraction transformed as a function of time was measured by optical microscopy. The results are shown in Fig. 12 where even small amounts of prestrain are shown to significantly increase the fraction transformed at a given time. This effect has been noted in a number of alloy systems [7, 8, 15, 23, 49, 50] and has been attributed to the increased driving force for the discontinuous transformation derived from the stored energy of plastic deformation. These data have been plotted as $\log \ln[1/(1-\zeta)]$ against $\log t$ and yield values of n which increase continuously from 0.8 to 1 with increasing prestrain. The increased rate of the discontinuous transformation relative to the continuously

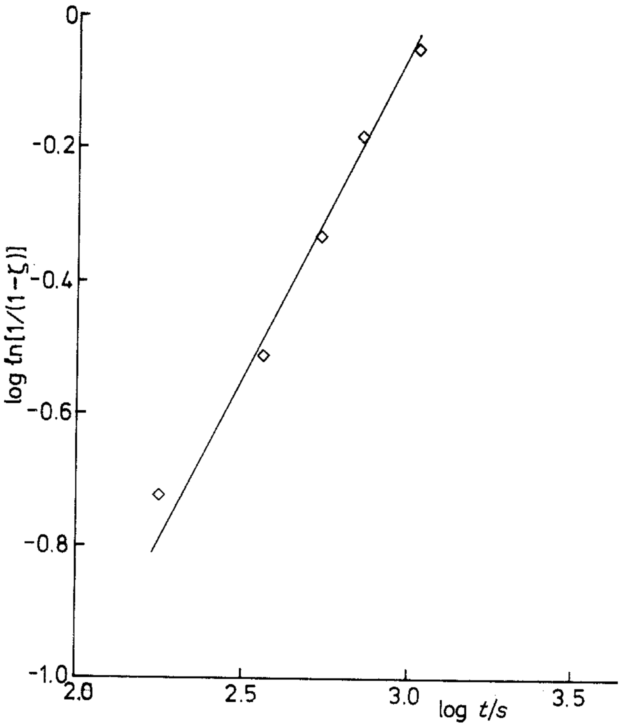

Figure 11 Reversion of discontinuous precipitate in Ni 38–8 at 1008 K.

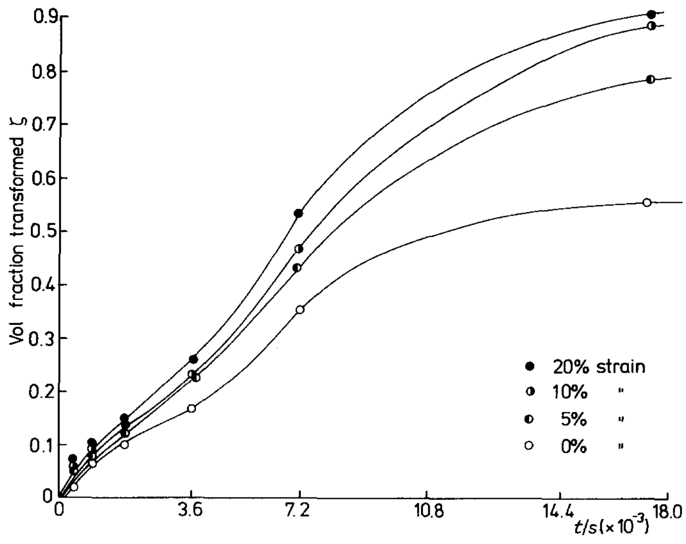

Figure 12 Fraction transformed as a function of time in Ni 38–8 aged at 750 K after various prestrains.

precipitated γ' particle coarsening rate clearly produces a more nearly constant growth rate and hence yields values of n nearer to 1.

4. Summary

Discontinuous precipitation has been observed to occur in all of the Ni–Co–Al alloys to some extent at temperatures below 973 K, the extent of precipitation decreasing with decreasing cobalt content in the alloys. Only in high cobalt alloys (greater than 38 at %) did the discontinuous transformation completely transform the specimens.

The discontinuous transformation product consisted of straight coherent γ' lamellae in a γ matrix at low ageing temperatures and short times. There was a specific cube–cube orientation relationship between γ and γ'. The constancy of the lamellar spacing was maintained during growth largely by branching of lamellae. Nucleation of discontinuous cells took place on both sides of a grain boundary resulting in an "S" type morphology.

At higher temperatures and longer ageing times, continuous and discontinuous coarsening of the discontinuous transformation product produced a coarse less regular γ/γ' structure with an average plate spacing of approximately 10 times that of the regular structure. The discontinuous and continuous coarsening processes were competitive, with the extent of the discontinuous coarsening reaction being limited to a maximum of 25% transformation as a result of continuous coarsening ahead of the moving reaction front. Discontinuous coarsening occurred by nucleation on both sides of a boundary producing an "S" type morphology which developed into double seams of material on either side of the boundary. Continuous coarsening occurred by the dissolution of the smaller lamellae, particularly at lamella faults and terminations, with concomitant thickening of the coarser lamellae.

The overall kinetics of the discontinuous transformation were affected by the continuous precipitation of γ' particles ahead of the discontinuous reaction fronts, the coarsening of these particles as a function of time stopping the transformation in low cobalt alloys and in high cobalt alloys at high tempertures. It is suggested that even in alloys which transform to completion by the discontinuous reaction that the growth rate varies with time resulting in "apparent" Avrami exponents $n < 1$. This suggestion is supported by the fact that in dissolution experiments, when the backward movement of the discontinuous reaction fronts is unaffected by the presence of continuous γ' precipitates, values of $n = 1$ are obtained. Further support to this argument is given by the fact that if the driving force for transformation is increased by prestrain, values of $n = 1$ are also obtained, in this case the increased rate of transformation making coarsening ahead of the reaction front less significant. It is clear that the major effect of cobalt in these alloys is to increase the propensity for discontinuous transformation. This effect is largely due to the decrease in rate of continuous precipitation and coarsening as a result of the increased partitioning of cobalt between γ and γ' in high cobalt alloys. However an effect of cobalt on boundary mobility cannot be dismissed.

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