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Precipitation study of heat-treated Incoloy 825 by scanning electron microscopy

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The Ni–Fe–Cr system forms the basis of a number of commercial alloys which have various applications in the chemical and nuclear industries because of their good corrosion resistance and high strength at elevated temperatures. Incoloy 825 is an Ni–Fe–Cr alloy with additions of Mo, Cu and Ti. It has applications in critically important components of the industries [1–3]. The mechanisms of sensitization and stabilization of this alloy against corrosion attack have been studied [4–6]. Sensitization in this alloy is shown to be due to the depletion of Cr because of formation of $M_{23}C_6$ precipitates at the grain boundaries and the alloy can be stabilized by the prevention of this phenomenon. However, no detailed study of precipitation in Incoloy 825 has been reported. The results of an investigation of precipitation in heat-treated Incoloy 825 using scanning electron microscopy (SEM) are presented in this letter.

The nominal composition of the alloy obtained from the commercial sources is given in Table I. Samples (1 cm × 1 cm or 1 cm in diameter) were cut from a sheet of thickness 1 mm. They were solution-treated at 1200 °C for 1 h. These samples were then aged at 870 °C for various intervals in the range 1–264 h. All of the heat treatments were done in air and the oxidation layer on the surface was removed by grinding and polishing on a lapping machine. The polished samples were etched by swabbing (for 20 s) with a 2:2:1:1 solution of glycerine, HCl, HNO_3 and H_2O_2 in order to reveal the grain boundaries. Fresh solution was used for every sample. A Jeol JSM-35CF SEM was used to study the microstructure of the samples, and elemental microanalysis was done using a Link energy-dispersive system. Samples were examined in three conditions: mill-annealed (i.e. as-received), solution-treated and aged. Microanalysis of the particles extracted in the form of carbon replicas was also carried out. The size distribution of the precipitates was determined by measuring the area of a number of precipitates using enlarged micrographs of suitable size.

In the mill-annealed condition, examination in the SEM showed the presence of certain particles (Fig. 1) within the grains. Very few of them were observed on the grain boundaries. Most of these precipitates have a definite geometrical shape, as is clear from Fig. 1. Microanalysis of these precipitates showed them to be rich in Ti and Cr. Microanalysis of the extracted particles revealed that there are three types of particles with Cr:Ti ratio of 1:1, Cr:Ti ration of 10:1, and Ti content around 90–95% and Cr about 2% (the rest being other elements such as Ni). Ageing does not appear to have any effect on the shape, size or composition of these precipitates. Solution-treatment at 1200 °C for 1 h does not have any appreciable effect on these precipitates.

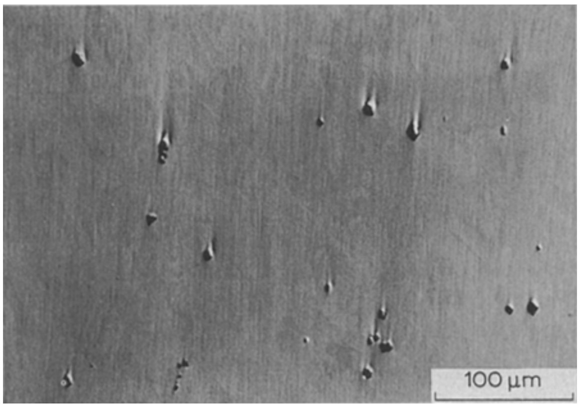

Figure 1 Ti-rich precipitates in as-received samples.

In the samples aged at 870 °C for various intervals, precipitates were observed at the grain boundaries as shown in Fig. 2. No such precipitates were observed in the mill-annealed and solution-treated

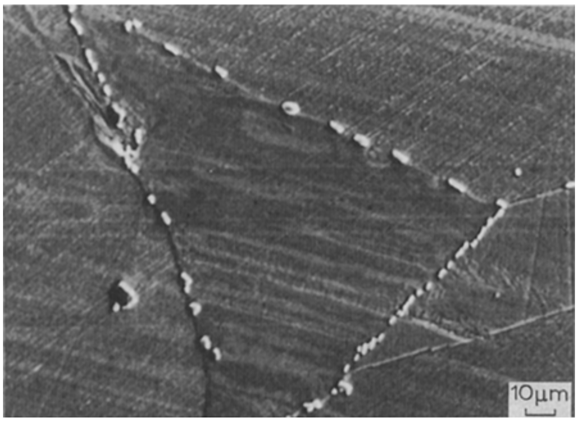

Figure 2 Cr–Mo-rich precipitates in aged samples.

TABLE I Nominal composition of Incoloy 825 (wt %)

Ni	Fe	Cr	Mo	Cu	Ti	Co	Si	Al	Mn	C	S
39.4	31.0	22.2	3.3	2.0	0.80	0.60	0.22	0.16	0.13	0.09	0.01

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but unaged samples. These precipitates are most probably $M_{23}C_6$, as it has been reported [7] that in nickel-base superalloys $M_{23}C_6$ are usually formed at grain boundaries. As shown in Fig. 2, these precipitates have different morphologies. Most have their long axis parallel to the boundary, whereas a few are elongated in a direction normal to the boundary. Some precipitates are observed to grow in both directions.

Elemental microanalysis revealed two types of precipitates in the aged samples. Precipitates of the first type had about 74% Cr and 10% Mo, whereas those of the second type had about 45% Cr and 13% Mo. The composition of such precipitates is given in Table II. Ageing does not appear to have any effect on the composition of these precipitates. The presence of two types of precipitates was confirmed by the application of the Lorimer and Cliff technique [8] to the elemental composition of these precipitates.

Three types of grain boundaries can be identified in Fig. 2: those with many precipitates of small size, some with fewer but bigger precipitates and others with no precipitates at all. Variation of the precipitate distribution from boundary to boundary is expected because grain boundary precipitation has been shown to be affected by the precipitate–matrix interface, inclination and misorientation of the boundary, the orientation of the boundary plane with respect to the habit plane of the precipitate [9]. Twin boundaries were observed to be free of precipitates (Fig. 3), because twins have a perfect coincidence relationship and precipitation is retarded on boundaries close to coincident-side positions [10].

Fig. 4 shows a plot of number density as a function of the size of precipitates. It shows that the minimum size of the precipitates remains almost constant, indicating that nucleation does not cease even after 264 h ageing. It is clear that the number of precipitates of bigger size increases initially with increasing ageing time, indicating an increasing growth rate. However, a plot of the maximum precipitate size as a function of the ageing time (Fig. 5) shows that the trend in the increase of maximum size ceases after about 100 h. A further increase in size may be hindered by the fact that nucleation is continuing. Fig. 6 shows an interesting feature of grain boundary

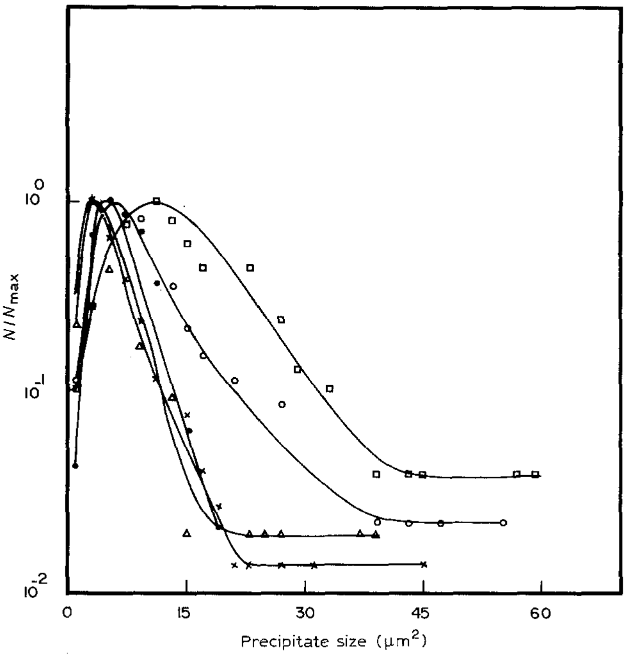

Figure 4 Number density as a function of precipitate size: ageing time (●) 20, (×) 96, (△) 120, (○) 168 and (□) 264 h.

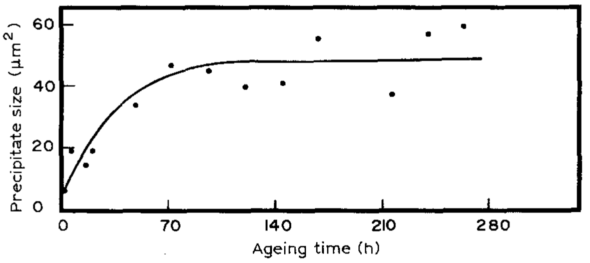

Figure 5 Maximum size of precipitates as a function of ageing time.

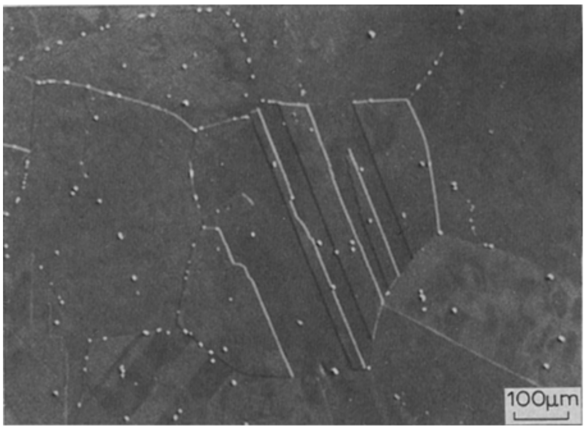

Figure 3 Lack of precipitation at twin boundaries.

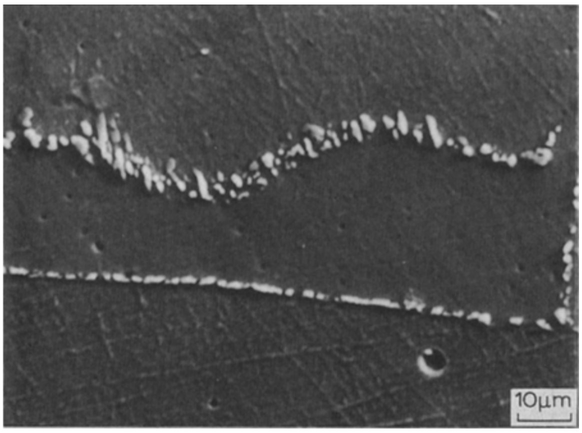

Figure 6 Grain boundary migration.

TABLE II Composition (wt %) of precipitates in the sample aged for 240 h

Precipitate	Cr	Mo	Fe	Ni
Type I	74.8 ± 1.9	10.7 ± 1.9	8.2 ± 2.4	6.1 ± 1.5
Type II	44.9 ± 3	12.3 ± 1.8	26 ± 1.5	16.7 ± 2.8

precipitation. It indicates grain boundary migration which may have been induced by precipitates or by other factors such as grain growth [9]. It is not possible to identify the actual cause of the boundary movement from the present results.

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