LETTER

THE EFFECT OF YTTRIUM ION IMPLANTATION ON THE SULPHIDATION OF INCOLOY 800H

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Previous work has shown the beneficial effect of yttrium implantation on the oxidation behaviour of chromium oxide forming alloys. Because of the limited applicability of Fe–Cr–Ni alloys in sulphur containing gases the effect of yttrium implantation on the sulphidation of Incoloy 800H was studied. In short time tests a significant improvement was observed upon implantation of about 10^{16} yttrium ions/cm^2 if an oxidizing treatment was applied before exposition to the sulphiding environment. The explanation of this effect is probably similar to that proposed in the literature for the improved oxidation resistance.

Several investigations have been reported concerning the influence of ion implantation on the oxidation of various metals and alloys [1–7]. In some cases no influence or even an acceleration of the oxidation has been observed [7], in others a benificial effect has been reported [2–6]. In particular, Bennett and coworkers [2,3,6] and Pivin and Roques-Carmes [4] have shown that yttrium implantation leads to an improved oxidation resistance of chromium oxide forming alloys. The parabolic rate constant at 1273 K was decreased by about 60% at an implantation dose of 9×10^{15} Y ions/cm^2.

This improved oxidation resistance is attributed by these authors to the incorporation of yttrium ions at the grain boundaries of the chromium oxide layer and thus hindering the outward diffusion of substrate metal ions.

As part of a research project directed at the improvement of the sulphidation resistance of stainless steels [8,9], it was decided to study the effect of yttrium implantation also. In this Letter the first results obtained are reported.

From the Incoloy 800H bar material rectangular specimens of $10 \times 8 \times 3$ mm^3 were machined after heat treatment of the bar at 1100°C, followed by quenching in water. The specimens were ground on emery paper (final pass 800 grit) followed by polishing with diamond paste (1 μm) and Al$_2$O$_3$ (0.05 μm). Finally they were cleaned ultrasonically in ethanol.

Direct implantation of yttrium ions was performed on the two largest sides (10 × 8 mm each) of the specimens, using the 110 keV implantation facility at
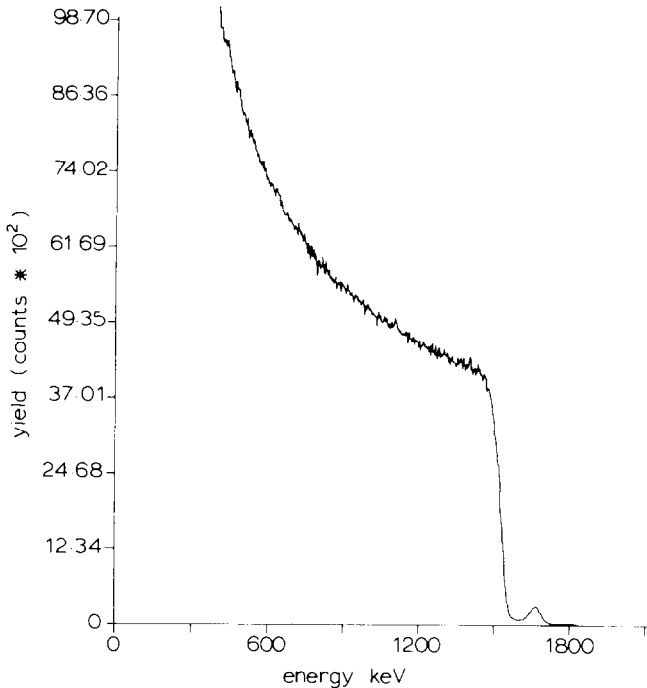
Fig. 1. Rutherford backscattering profile of directly implanted specimen.

the University of Groningen with $Y_2O_3 + CCl_4$ as starting material. The current density applied during the implantations was 2 $\mu A/cm^2$ and the totally implanted dose 10^{16} Y ions/cm^2.

The implanted specimens were investigated by means of Rutherford backscattering at the University of Utrecht.

Thermogravimetric measurements, both of oxidation and of sulphidation, were performed using a Cahn-1000 electrical thermobalance.

A typical Rutherford backscattering profile is shown in fig. 1. From this the dose was calculated to be 9×10^{15} Y ions/cm^2, in good agreement with what was calculated from the current density. The average implantation depth was found to be about 160 Å and the maximum concentration about 3 at%.

The oxidation of both unimplanted and implanted specimens was investigated by means of thermogravimetry in a mixture of 70 ml/min oxygen and 180 ml/min argon. After a non-parabolic start, the oxidation became parabolic after some hours. The parabolic rate constant of the implanted material was about 45% lower than that of the unimplanted material, in reasonable agreement with the results of Pivin and Roques-Carmes [4].

Table 1
Weight gain (in mg/cm^2) during sulphidation

Time	Unimplanted	Implanted	Preoxidized	
			Unimplanted	Implanted [a]
2	1.2	1.2	0.0	0.0
3	1.9	2.0	0.0	0.0
5	2.1	2.1	0.01	0.0
10	--	--	0.12	0.0
20	--	--	0.32	0.0
30	--	. --	0.55	0.0
40	--	--	0.91	0.0
50	--	--	1.32	0.0

[a] Corrected for the sulphidation of the unimplanted side surfaces.

Sulphidation was performed at 560°C in a gas mixture consisting of 78.3% Ar, 20.6% H_2 and 1.1% H_2S and containing approximately 100 ppm O_2 as an impurity. The calculated $p_{S_2} = 1.8 \times 10^{-9}$ bar and $p_{O_2} = 10^{-32}$ bar. The sulphidation was performed both directly after implantation and after pre-oxidation at 1020°C in the same gas mixture used for the oxidation experiments. This preoxidation was also performed in the thermobalance in order to obtain, as far as possible, the same oxide thickness before implantation. On the unimplanted samples the thickness obtained was 1.7 μm after 24 min oxidation at 1020°C, on the implanted samples 1.2 μm after 45 min. In table 1 the main results are collected.

It is clear that, at least for the times used in this investigation, implantation combined with preoxidation gives complete protection against sulphidation. Microscopic investigation at a magnification of 1600 × showed no trace of surface reaction on the implanted and preoxidized surfaces.

Because preoxidation is evidently necessary to obtain good protection, it seems reasonable to suppose that the mechanism of the protection is similar to that proposed in the literature for oxidation [2–4, 6], namely that the yttrium ions are taken up in the chromium oxide layer and hinder the diffusion of metal ions through the product layer.

At the moment further experiments are being performed in which longer exposure times and higher implantation doses are being studied.

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