

Letters

Scanning and transmission electron microscopy of Ni-Mo alloys exposed to HBr

Ni-rich alloys of the system Ni-Mo, Ni-W and of Ni-Ta are interesting structural materials for chemical plants owing to their resistance to non-oxidizing acids.

For the system Ni-Mo, Pavlov and Svistunova [1] have shown by weight determination that the Ni-rich alloys are susceptible to corrosion by boiling HCl after heating at temperatures between 600 and 900°C. Within this temperature range the Ni-rich Ni-Mo alloys undergo order-disorder transitions. Extensive TEM studies were recently made of the substructures which form during these order-disorder processes [2-5].

In order to get some detailed information concerning the corrosion of Ni-rich Ni-Mo alloys in non-oxidizing acids, an attempt was

therefore made to correlate the results of weight determination with the results of (a) SEM studies of the surface structure before and after corrosion and (b) TEM studies of the internal structure. The Ni-Mo alloys were suitably chosen to contain 20 to 25 at. % Mo. Samples of these alloys were first annealed at different temperatures and then exposed to 48% HBr at 126°C for up to 24 h.

The weight loss of the samples after exposure to HBr was generally small ($\leq 0.5 \text{ mg cm}^{-2}$ for 24 h). The loss, however, was definitely dependent on the thermal pretreatment.

A relatively large loss was observed for short-range ordered samples quenched from the α -solid solution region. According to TEM studies of Okamoto and Thomas [5] the structure of short-range ordered Ni-Mo samples quenched from the α -region, consists of microdomains exhibiting Mo-concentration fluctuations. The present SEM studies of quenched,

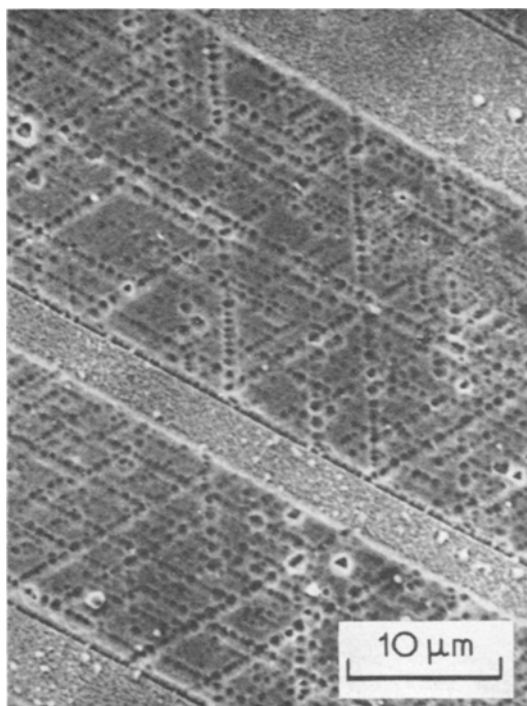


Figure 1 SEM micrograph of the surface of short-range ordered $\alpha(\text{Ni}_3\text{Mo})$, quenched from 1200°C and subsequently exposed to HBr for 6 h. Twinned region, etch pits on (111) planes marking dislocations.



Figure 2 SEM micrograph of the surface of long-range ordered $\beta(\text{Ni}_4\text{Mo})$ after exposure to HBr for 16 h. Regular corrosion pattern can be seen.

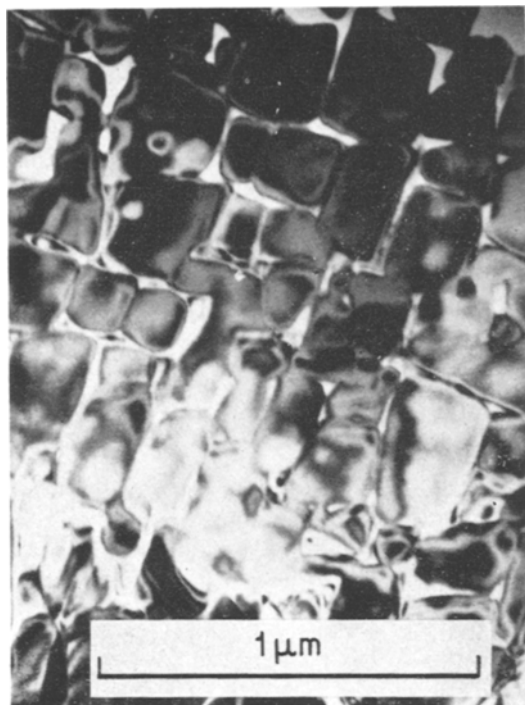


Figure 3 TEM micrograph of the internal structure of the specimen shown in Fig. 3. Domain structure present.

short-range ordered samples exposed to HBr, revealed a uniform attack of the grains and also a selective attack of some boundaries. Sometimes deformed regions were also observed in the as-quenched specimens through SEM and TEM. In such regions, after suitably chosen exposure times, the close-packed planes were etched by HBr (Fig. 1).

The corrosive attack of long-range ordered Ni-rich Ni-Mo samples depended on whether a domain structure was present in the original α -grains. The smallest loss ($\sim 0.15 \text{ mg cm}^{-2}$ for 24 h) was found for samples containing the ordered phase β (Ni_4Mo) or γ (Ni_3Mo) not

exhibiting any domain structure. In the grains of such specimens no relevant corrosion pattern was observed by SEM except that some grain boundaries were attacked. On the other hand, any long-range ordered specimen containing a domain structure in the grains showed an enhanced loss and specific corrosion patterns, particularly for the phase β (Ni_4Mo). Figs. 2 and 3 demonstrate the surface and internal structure of a Ni_4Mo specimen quenched from 1100°C , long-range ordered at 850°C for 40 h and then exposed to HBr. By comparing the two micrographs one is led to conclude that the corrosion pattern visible in Fig. 2 is due to an attack on the numerous domain boundaries present in the specimen after the annealing treatment.

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Cooling-rates in splat-cooling

Being involved in splat-cooling studies and having utilized several different methods to determine the cooling-rate of the splat foils, we were quite interested in the recent exchange of opinions and ideas which appeared in the *Journal of Materials Science* [1, 2]. May we please add our thoughts?

Many attempts have been made to determine the cooling-rates achieved in splat-cooling. These

include theoretical calculations [3] as well as direct [4-6] and indirect [7, 8] measurements. Of all the rapid quenching techniques employed, the gun technique [4, 9] is probably the most difficult technique for measuring the cooling-rate. This is due to the wide range of splat thicknesses found (thicknesses may vary from $0.05 \mu\text{m}$ to over $25 \mu\text{m}$ in a single foil) and the variation of heat-transfer coefficients found from splat to splat. As shown by Ruhl [3], the cooling-rate varies inversely with the square of