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# THE AS-QUENCHED MICROSTRUCTURES OF RAPIDLY SOLIDIFIED

Fe-25wt%Ni

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## Abstract

Specimens of rapidly solidified Fe-25wt% Ni have been obtained by two-piston splat-quenching. The as-quenched microstructure varies from fully austenitic to almost completely martensitic. The microstructures have been studied by x-ray diffraction, scanning and transmission electron microscopy and x-ray microanalysis. The fully austenitic specimens reveal a fine grain size (1-5 $\mu$ m) with a high dislocation density of  $\sim 10^{11}$  dislocation lines  $\text{cm}^{-2}$ . There is evidence for the existence of sub-grain boundaries in some of the grains. In addition, two different martensitic

microstructures are observed. In the specimens which have partially transformed to martensite during quenching, the structure is lath martensite with fine internal twins present in some laths. However, in austenitic specimens which have been transformed by cooling in liquid nitrogen, the microstructure is lenticular plate martensite with the characteristic mid-rib. The different structures found in rapidly solidified Fe-25wt%Ni are discussed.

### Introduction

Although Rapid Solidification Processing (RSP) has produced interesting and often novel results in the area of amorphous metals, it has been used more recently as a means of studying more conventional materials and their phase transformations. In the field of ferrous alloys, RSP has been applied to a range of different compositions, from pure iron (1) through simple binary Fe-X alloys (2), to the more complex high speed tool steels (3,4). In all these cases, the main results of RSP have been shown to be (a) the refinement of the ferrite/austenite grain size and (b) either the complete solution of both carbon and alloying elements, or the fine homogeneous precipitation of carbides. The present investigation is into the effect of RSP upon binary alloy, Fe-25wt%Ni. A previous preliminary investigation (5) has shown that this alloy, when rapidly quenched from the melt, remains austenitic instead of transforming to lath martensite at an  $M_s$  temperature of  $\sim 140^\circ\text{C}$ . The aim of this study, therefore, is to examine in more detail the microstructure achieved through RSP, and compare this with the microstructure observed in the conventionally solution-treated and quenched alloy.

### Experimental Procedure

An alloy of Fe-25wt%Ni was prepared from  $\geq 99.99\%$  pure iron and nickel, by induction-melting in a recrystallised alumina crucible

under a dynamic argon atmosphere. The as-cast alloy was homogenised at 1100°C for 50 hours and specimens were taken from the homogenised ingot for rapid solidification. RSP was achieved by means of a two piston splat-quenching apparatus described in detail elsewhere (6). Individual specimens of 0.50-0.80 gram mass were levitation-melted in an argon environment and then allowed to fall under gravity until quenched between two copper pistons accelerated towards each other magnetically. The RSP foils produced were generally 60-100µms in thickness and 2-3cms in diameter. The effective cooling rate has been measured as  $\sim 10^7 \text{ Ks}^{-1}$  at the solidification temperature falling to  $\sim 10^5 \text{ Ks}^{-1}$  at 700°C (1). A combination of experimental techniques was used to examine and characterize the microstructure(s) of the RSP alloy. These included x-ray diffractometry, scanning and transmission electron microscopy (SEM, STEM and TEM) and x-ray microanalysis.

### Results

Rapid solidification processing of Fe-25wt%Ni resulted in a range of microstructures, from fully austenitic ( $\gamma$ ) to almost completely martensitic ( $\alpha'$ ). The former microstructure, as seen in Fig. 1, was a fine-grained solidification structure with an austenite grain size of between 1.0-5.0µms in diameter, and a dislocation density of  $\sim 10^{11}$  dislocation lines per sq.cm. This structure, reported in more detail elsewhere (7), consisted of  $\gamma$  grains separated mostly by grain-boundaries of  $\sim 7^\circ$ - $15^\circ$  misorientation, but occasionally by low-angle grain boundaries ( $<5^\circ$ ). In addition, some  $\gamma$  grains contained well defined subgrains separated by low-angle dislocation sub-boundaries. Although most RSP foils were fully austenitic, a few foils had at least partially transformed to martensite during quenching. The resulting microstructure,

Fig. 2, was identifiable as lath martensite. The laths were typically  $\sim 0.1$ - $0.2$  microns in width and  $1$ - $5.0$  microns length, parallel-sided, and separated generally by low-angle lath boundaries. However, there were areas observed in which laths within one packet were twin-related, and also many laths were found to contain internal twins. In virtually all areas examined, the packet size was equivalent to the prior austenite grain size (see Fig. 3). It was apparent that in the case of the fully austenitic foils, the  $M_s$  temperature had been depressed to below room temperature. Therefore, in an attempt to measure the new  $M_s$  temperature and to examine the subsequent martensitic microstructure, a series of cooling experiments were carried out on one fully austenitic foil, at temperatures less than ambient. The specimen was cooled from  $0^\circ\text{C}$  to  $-100^\circ\text{C}$  in steps of  $10$  degrees, and after each step, was allowed to warm up to room temperature. Once at this temperature, the specimen was examined by x-ray diffractometry to establish the phases present, and then cooled once more to the next temperature. This was repeated until the first signs of a bcc phase were detected. Although a little crude, this procedure was able to show that the  $M_s$  temperature was depressed by  $\sim 200$  degrees to a value of  $\sim -70^\circ\text{C}$ . The specimen was further cooled to liquid-nitrogen temperature, in an effort to completely transform the foil to martensite. However, even after  $24$  hours at this temperature, some austenite remained. The microstructure of the specimen was examined at different stages of the cooling, both by SEM and TEM, and examples of both types of microscopy are shown in Figs. 4 and 5 respectively. It is clear that in the cooled-and-transformed specimen, a different martensite transformation has occurred from that observed in the as-quenched-and-transformed specimens. The microstructure

was no longer lath but plate martensite, with lenticular plates and well-defined mid-ribs. The dimensions of this new martensite were observed to be 0.1-0.2 microns in width, and 0.4-0.5 $\mu$ m in length. In the conventional solution-treated and quenched alloy, only lath martensite is present (10).

### Discussion

RSP of Fe-25wt%Ni results in an as-quenched microstructure ranging from fully austenite to an austenite/martensite mixture. During the splat-quenching process, experimental parameters such as the specimen mass, power applied, and time held in levitation were kept as constant as possible. However it was more difficult to keep the contact position and the surface condition of the two pistons as reproducible. It is likely that this led to a variation in the deformation of the specimen, and in the thermal contact between the pistons and the molten droplet, and thus the heat flow away from the specimen. These differences in as-quenched microstructure are a direct result of a variation in the  $M_s$  temperature in the RSP foils. An austenite specimen was found to have a  $M_s$  of  $-70^{\circ}\text{C}$  whereas a partially transformed austenite/martensite specimen had a  $M_s$  of  $40^{\circ}\text{C}$  (8). These values are both significantly lower than the  $M_s$  temperature of the conventional alloy, which is  $\sim 140^{\circ}\text{C}$ . The cause of such a depression in the  $M_s$  is not fully known, but it is probably a combination of two effects: (i) the deformation produced by the squeezing action of the pistons upon the molten droplet/ solidified foil, and (ii) a small grain size resulting in a Hall-Petch strengthening of the austenite. The former effect is significant enough to produce a high dislocation density in the foils (high enough, in some cases, to result in the formation of dislocation sub-grains), and therefore work-harden the austenite. Both of these effects result in

in strengthening of the austenite which has been shown previously to lead to a lowering of the  $M_s$  temperature (9).

An interesting result of this investigation is the occurrence of both major types of martensite at the same alloy composition. Many ferrous systems, including Fe-Ni, exhibit both lath and plate martensite, but the former is usually present in dilute alloy compositions, whereas the latter forms at higher compositions. Previous studies have concluded that the main factors affecting the transition from lath to plate are alloy composition and/or transition temperature (10). In the present case, it is clear that the  $M_s$  temperature and not the composition is dictating the transition. All foils were obtained from the same homogenised ingot, and the ingot composition was confirmed by x-ray analysis as being Fe-25wt%Ni. Moreover, representative foils of the two types of martensite were examined using STEM/X-ray Microanalysis; no segregation was detected and both specimens had the same composition as the bulk ingot. The manner in which the  $M_s$  temperature influences the transition is by determining which transformation mechanism (either slip or twin-type) is appropriate for the composition and temperature conditions experienced. Therefore, any factor which can effect the  $M_s$  temperature will effectively control the final martensite morphology of an alloy. Such a factor could well be the strength of the parent phase. Increasing the austenite strength by (i) solid-solution strengthening (a compositional effect), (ii) decreasing the grain size (a Hall-Petch effect) or by (iii) work-hardening, will enable the austenite to resist the shears needed to nucleate martensite and thus lead to a lowering of the  $M_s$  temperature.

### Conclusions

Rapid Solidification Processing of Fe-25wt%Ni results in the following:

- (i) A fine-grain austenite microstructure in comparison to lath martensite normally observed in the conventional alloy.
- (ii) A depression of  $M_s$  by as much as 200°C. This is almost certainly caused by austenite strengthening through a combination of Hall-Petch strengthening and work-hardening and
- (iii) The appearance of both major types of martensite in specimens of the same composition. The transition from lath to plate is found to be dependent only on the  $M_s$  temperature.

#### Acknowledgements

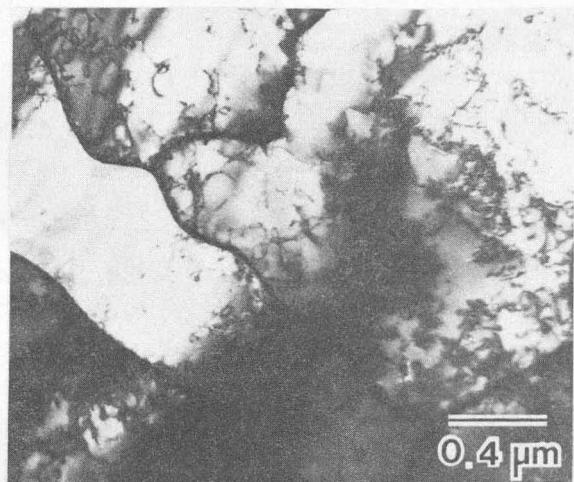
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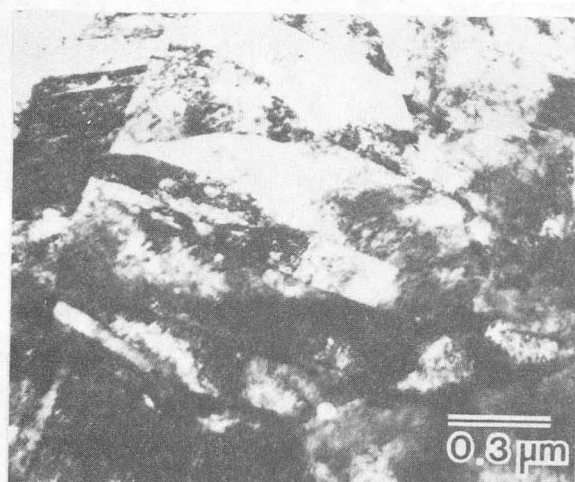
Figure Captions

- Fig. 1. The austenite microstructure of RSP Fe-25wt%Ni
- Fig. 2. The lath martensite microstructure of RSP Fe-25wt%Ni; (a) B.F., (b) D.F. from an  $(002)_T$  reflection
- Fig. 3. The lath martensite microstructure of RSP Fe-25wt%Ni
- Fig. 4. An SEM micrograph of plate martensite in RSP Fe-25wt%Ni
- Fig. 5. The plate martensite microstructure of RSP Fe-25wt%Ni



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Figure 1



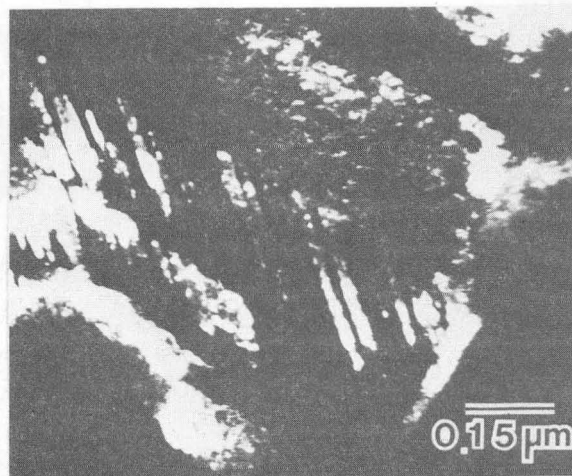
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Figure 3



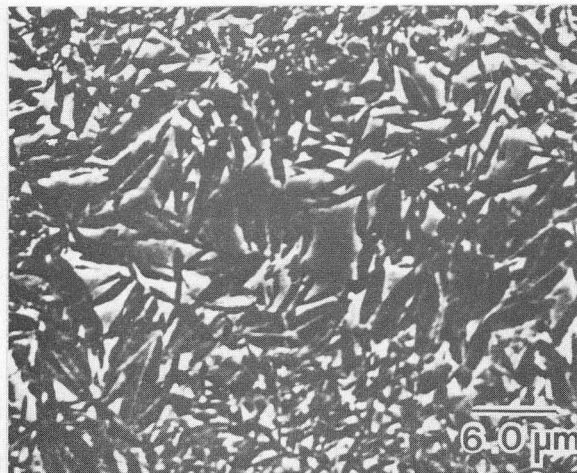
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Figure 2a



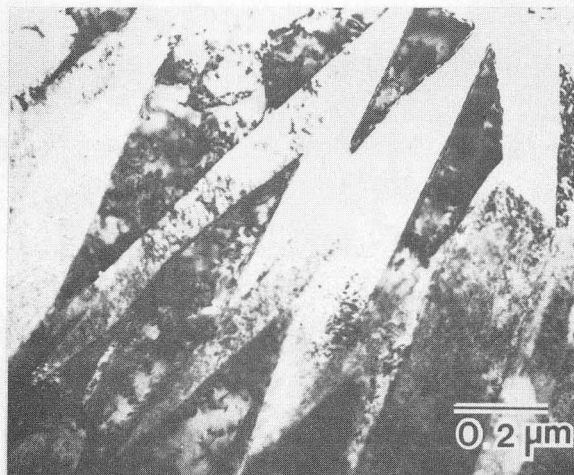
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Figure 2b



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Figure 4



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Figure 5

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