

On mica cleavages, bismuth forms three orientations, (001), (011) and (012). At temperatures above 130°C the films are purely (001) oriented [5]. Antimony films on mica are made up of two orientations, (001) and (011) when deposited at room temperature. An increase in the substrate temperature up to 80°C results in a predominant (001) orientation. However the (011) orientation is still present. Further increase in the temperature results in the formation of polycrystalline deposits.

The initial stages of growth of bismuth and antimony films and their orientation on sodium chloride and mica cleavages differ considerably. The crystallinity of bismuth films improves continuously with increase in temperature whereas antimony films deposited at a comparatively lower substrate temperature are better oriented than those deposited at higher temperatures. Similar results have been reported by Mojeiko *et al.* [6] and they attribute this to the changes in the size of the critical nuclei and also the number density of the nuclei. It seems more probable that the high mobility of the adatoms on the substrate surface is responsible for the polycrystallinity of thicker deposits as the initially oriented nuclei grow and the film passes from the nucleation stage to the growth stage.

The nature of growth and orientation of bismuth films seems to be different from that of antimony films. Increase in the deposition temperature improves the crystallinity of bismuth films whereas the antimony films do not show any improvement when deposited on sodium chloride substrates. However, antimony films show a better orientation on mica at 80°C which is destroyed

with further increase in temperature. As the film passes from the nucleation stage to the growth stage, the initially well-oriented nuclei tend to become polycrystalline because of the high mobility of the adatoms on the substrate surface.

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### *Deformation properties of austenitic Fe-Ni-C alloy single crystals*

Although numerous studies have been made to investigate the properties of austenitic single crystals in some ferrous alloys [1-6], the main purpose of these studies was to clarify the mechanism of martensite formation in the single crystal state. However no work has yet been reported on the deformation of austenitic Fe-Ni-C single crystals and the effect of martensite formation

on the mechanical properties of the material, probably because of the practical difficulties in obtaining large crystals. In the present investigation it was thought worthwhile therefore to study some deformation properties of austenitic single crystals in two Fe-Ni-C alloys of different composition.

The previous work on the deformation of polycrystalline Fe-Ni-C alloys [7, 8] revealed that the mechanical properties of the material were considerably changed after the austenite

to martensite transformation, as a function of the martensite volume fractions. This change could not be determined easily because of the varying grain size in the polycrystalline samples. Therefore, studies with single austenite crystals might be more convenient to clarify the mechanism.

Two alloys of compositions Fe–26.4 wt% Ni–0.24 wt% C and Fe–24 wt% Ni–0.45 wt% C were supplied by the United Steel Co Ltd, UK, in the form of 10 kg ingots. Both alloys had sub-zero austenite to martensite transformation temperatures ( $M_s$ ). Single austenite crystals were obtained by spark cutting large grains from recrystallized samples. These samples were prepared from the bulk material in the form of cylinders of 2 cm diameter and 1 cm length and were cold rolled between 3% and 5% and annealed for 6 hours at 1300°C in a vacuum of  $10^{-5}$  torr. Deformation of the material prior to the annealing process was found to be an effective way of increasing the austenite grain size and by using this method recrystallized grains were obtained with diameters greater than 6 mm.

A compression test machine which had a maximum capacity of 1000 kg was used for the compression tests. The load cell was calibrated to 1% prior to each test and a cross-head speed of  $105 \mu\text{m min}^{-1}$  was used for all experiments. Compression specimens were first cut with a wire spark cutter then held in special steel jigs and ground to  $2 \text{ mm} \times 2 \text{ mm} \times 5 \text{ mm}$  with silicon carbide papers. The load cell output of the compression test machine was fed into the pen recorder and measured at 50 kg steps.

Specimens for microscopical examinations were electropolished in 5% perchloric acid–methanol

solution and observed in a Cambridge Stereoscan MK1 scanning electron microscope. The same instrument was also used to determine crystallographic orientations of austenite single crystals by means of selected area channelling patterns.

The austenite phase in Fe–Ni–C alloys has a face-centred cubic crystal structure and stress–strain curves of this structure in metal crystals generally exhibit three different stages. However, under some conditions only one or two stages were found. The stress–strain behaviour of the austenitic single crystals of two Fe–Ni–C alloys was first examined at room temperature with the crystals having compression axes in the central region of the unit triangle. The stress–strain curves obtained are shown in Fig. 1. Since strength of the single crystal materials is orientation-dependent both samples were prepared with the same axis parallel to the compression direction and as seen in the figure, the higher carbon alloy single crystal was usually found to be “harder”.

More experiments were carried out to clarify the effect of the direction of applied stress on the mechanical behaviour. Fig. 2 shows the stress–strain curves of four austenitic crystals of the Fe–24 wt% Ni–0.45 wt% C alloy which had different compression directions. Examination of the stress–strain curves reveals that Fe–Ni–C single crystals also exhibit the typical orientation dependence of fcc structures, which have a relatively “soft” strength for the orientations close to the  $\{110\}$  pole.

Compression tests on the transformed austenite single crystals were carried out with two crystallographically identical samples of each alloy after transformation to martensite, at  $M_s$  and at

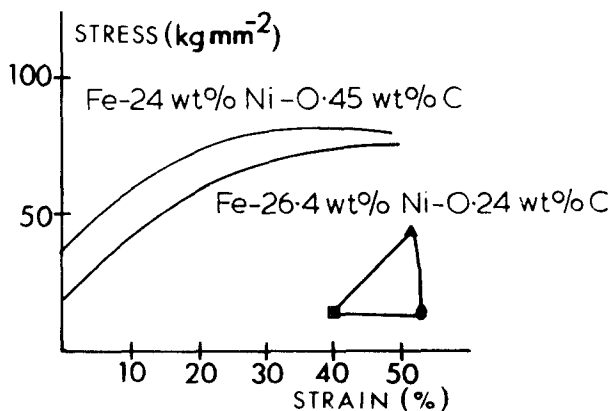


Figure 1 Stress–strain curves of austenitic Fe–26.4 wt% Ni–0.24 wt% C and Fe–24 wt% Ni–0.45 wt% C alloy single crystals. The compression direction of the crystals is shown in the unit triangle.

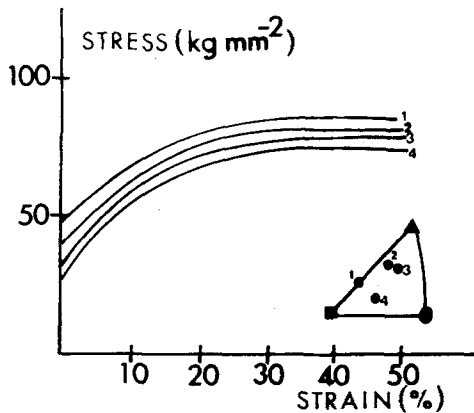


Figure 2 Stress-strain curves of four Fe-24 wt% Ni-0.45 wt% C austenite single crystals and corresponding compression directions.

liquid nitrogen temperature, to compare the effects of different martensite volume fractions upon the mechanical properties. Fig. 3 is a scanning electron micrograph of the martensite plates formed in the Fe-24 wt% Ni-0.45 wt% C alloy crystal after transformation at the  $M_s$  ( $-50^\circ\text{C}$ ) temperature. The martensite volume fractions were determined for this alloy by metallographic methods and found to be, relatively,  $\sim 20\%$  and  $80\%$ , after transformation, at the  $M_s$  and liquid nitrogen temperatures. Stress-strain curves of the single austenite crystals of both Fe-Ni-C alloys tested at room temperature are given in Figs. 4 and 5. Orientations of the single crystals are also shown in the figures. Examination of the curves reveals that, for example, for the Fe-24 wt% Ni-0.45 wt% C alloy the determined increase in the martensite volume fraction due to altering the transformation temperature from  $M_s$  to liquid nitrogen results in a change of about 50% in the stress at the strains of 10% and 20%.

The slip geometry of fcc materials is well documented in the literature, but as mentioned above, no previous work has been reported on the properties of austenitic Fe-Ni-C single crystals. Therefore, the nature of the slip in the single crystals of the alloy Fe-26.4 wt% Ni-0.24 wt% C was also examined in the present study. Austenite single crystals were prepared in various orientations and were all deformed by compression at room temperature. The operative slip planes were observed after about 5% axial strain. The slip planes and directions were determined using an

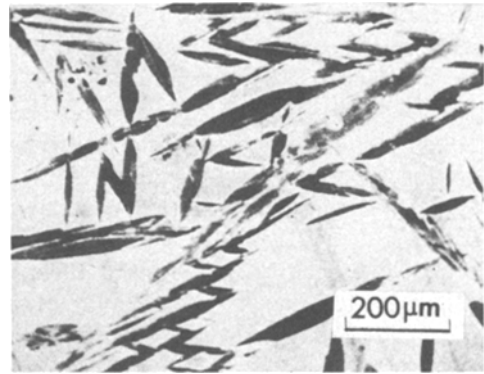


Figure 3 Scanning electron micrograph of martensite plates formed in an Fe-24 wt% Ni-0.45 wt% C single crystal at the  $M_s$ .

electron channelling pattern technique. Examination of all single crystals showed that the slip system is  $\{111\} \langle 110 \rangle$  as expected. Fig. 6 shows the slip bands of a single crystal compressed in the  $\langle 321 \rangle$  direction.

More than one operative slip system was found for the samples deformed parallel to the  $\langle 211 \rangle$  and  $\langle 111 \rangle$  directions which was also expected as it is a normal characteristic of the slip geometry in fcc materials. The sample compressed parallel to the  $\langle 211 \rangle$  direction exhibited two operative slip systems whilst that compressed parallel to the  $\langle 111 \rangle$  direction exhibited three. Fig. 7 is a scanning electron micrograph of the latter sample showing slip bands formed in the three different systems.

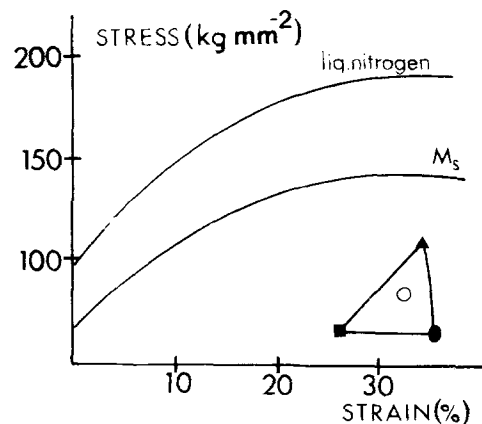


Figure 4 Stress-strain curves of Fe-26.4 wt% Ni-0.24 wt% C alloy single crystals transformed to martensite at  $M_s$  and liquid nitrogen temperatures, and the compression direction of the crystals.

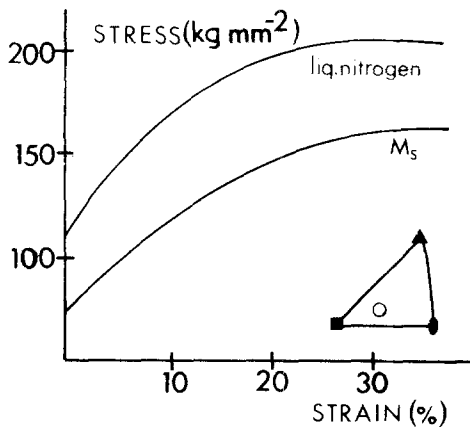


Figure 5 Stress-strain curves of Fe-24 wt% Ni-0.45 wt% C alloy single crystals transformed to martensite at  $M_s$  and liquid nitrogen temperatures, and the compression direction of the crystals.

Although it is more obvious in first-stage hardening, generally all parts of the stress-strain curves of fcc metal single crystals show changes as the direction of the applied stress is varied, so that the mechanical properties of the sample can be relatively "soft" or "hard". Compression axes parallel to the  $[100]$ - $[111]$  side of the stereographic triangle exhibit "harder" mechanical properties than the other orientations of fcc metal crystals. Since this structure has four and three operative slip systems on the  $\{100\}$  and  $\{111\}$  poles, it can be predicted that the "hard" nature of an individual single crystal is related to the orientation in which more slip systems operate.

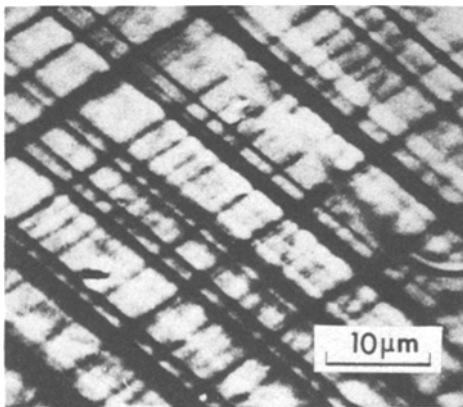


Figure 7 Scanning electron micrograph of slip bands formed in three different systems of an austenitic Fe-26.4 wt% Ni-0.24 wt% C alloy single crystal.

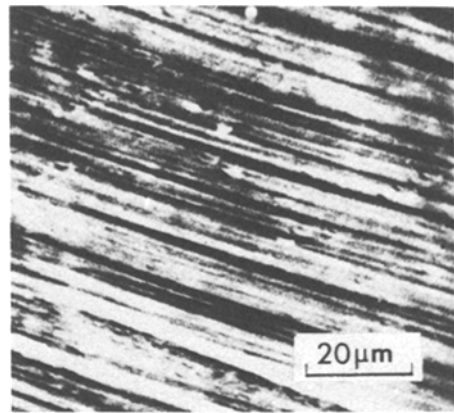


Figure 6 Scanning electron micrograph of slip bands in an austenitic Fe-26.4 wt% Ni-0.24 wt% C alloy single crystal.

Naturally composite effects are involved in the stress-strain behaviour of the transformed samples and these depend on both the size and the configuration of the martensite plates in the single austenite crystals and relatively "soft" strength of the matrix [7, 8]. The austenite grain size has a significant effect on the formation of the plates. Brook and Entwisle [9] reported that although there is a similarity between fine and coarse-grained materials, because of the formation of the plates across the full width of the austenite grains in the early stages of the transformation in fine-grained materials, plates in these materials are relatively much broader and have more volume fraction in the austenite matrix. Therefore, for the single austenite crystals it may be expected that the strength increase caused by the martensite is also smaller than that in polycrystalline materials, in addition to the lower austenite strength. The results obtained in the present study are in agreement with these predictions. A comparison of the results with those of the early work which has been done by Maxwell *et al.* [7] and Durlu [8] with polycrystalline Fe-Ni-C samples of almost the same compositions shows that the strength of the material is smaller in the single crystalline state for both the untransformed and transformed condition.

The first stage of the stress-strain curves was absent in all the tests carried out during the work. It is well known that plastic deformation of austenite at room temperature causes the formation

of strain-induced martensite and the presence of this martensite changes the mechanical properties of Fe-Ni-C austenites [7, 8]. Observations on the compression samples after the tests showed the existence of strain-induced martensite with various volume fractions. The martensite starts forming as the plastic deformation of the austenite begins. Therefore the absence of first-stage hardening might be due to the formation of strain-induced martensite.

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### *An electron microscopic study of crystalline turquoise*

Turquoise is generally considered to be a non-crystalline, massive, hydrated mineral composed of copper, aluminium, and phosphorous in oxide combinations, and isomorphous with chalcocyanite. The generally accepted composition is given as either  $\text{CuO} \cdot 3\text{Al}_2\text{O}_3 \cdot 2\text{P}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$ ,  $\text{CuOH} \cdot 6[\text{Al}(\text{OH})_2] \cdot \text{H}_5 \cdot (\text{PO}_4)_4$ , or  $\text{CuAl}_6(\text{PO}_4)_4(\text{OH})_8 \cdot 5\text{H}_2\text{O}$  [1-3]. As far as can be determined, the only crystalline turquoise subjected to mineralogical analysis were samples from Campbell County, Virginia [2, 3]. This turquoise consisting of thin layers of turquoise crystals cementing fragments of glassy quartz, was composed of individual crystals rarely larger than 0.3 mm in length, and considerably thinner. Schaller [2] determined these crystals to be triclinic because of their isomorphism with chalcocyanite, and using crystals which were at most 0.4 mm wide and little more than 0.1 mm thick. These crystals were colourless to bright blue in massive form, and have been repeatedly referred to as being minute and very rare [4]. It appears, in fact, that none have been

found outside Campbell County, Virginia, and there are no indications that crystals of turquoise have, in fact, been photographed, or studied in any detail since the very early and original crystallographic investigations by Schaller [2] and the more recent X-ray work [3]. Erd *et al.* [5] have also discussed the structure of turquoise in connection with faustite, a zinc isomorph.

In the present study, chalk turquoise from Nogales, Mexico was observed to be crystalline and the crystal structure was investigated by scanning and transmission electron microscopy. Individual crystals were examined and selected-area electron diffraction patterns obtained.

Large samples (10<sup>2</sup> g or greater) of Nogales, Mexico chalk turquoise were randomly broken to expose fresh interior structural features. The freshly exposed surfaces were a very pale blue (a blue-white), with an appearance and texture similar to plaster-of-paris. Small exposed samples not larger than 1 cm were mounted on 1.25 cm diameter cylindrical aluminium supports for observation in a scanning electron microscope using a silver-emulsion conducting paste. The mounted specimens were then coated by sputtering approxi-