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ULTRASONIC ATTENUATION AND VELOCITY CHANGES DURING THE FCC-HCP MARTENSITIC TRANSFORMATION IN COBALT-NICKEL

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Abstract. - Measurements were made to search for evidence of widening of extended dislocations as the transformation temperature is approached in a single crystal Co$_{68}$Ni$_{32}$ alloy. Specimens were prestressed plastically in shear to introduce dislocations primarily on one slip system. During transformation, measurements were made of longitudinal and shear strain as well as velocity and attenuation at 10 MHz. The sound waves were polarized to excite the "optical" mode of vibration of the two partial dislocations using a (111) [110] polarization shear wave. Pre-transformation changes were found which are consistent with a simple model for the effect.

1. Introduction. - A simple dislocation model for the FCC-HCP martensitic phase transformation predicts an ultrasonic velocity decrease and attenuation increase as a precursor to the transformation. We looked for the effect in the cobalt-nickel system, which has been studied extensively.

By using the alloy Co$_{68}$Ni$_{32}$ the transformation temperature is brought down to near room temperature with $M_s \approx -10^\circ$C and $A_s \approx 110^\circ$C. In addition de Lamotte and Alstetter showed that by prestressing samples of the alloy they could obtain single crystal to single crystal transformation in a .1 inch by 1 inch rod. Weston and Granato used this technique to obtain a single crystal cubic of hcp phase large enough (about 1 cm$^3$) to obtain complete sets of elastic constants in both phases.

2. Experimental Procedure. - Several samples of Co$_{38}$Ni$_{32}$ were cut to approximate dimensions [111] = 4 mm, [110] $\approx$ [112] $\approx$ 12 mm from a single crystal ingot. The (111) faces were polished flat and parallel for good ultrasonic pulse reflection. Samples were annealed at 930°C for 16 hours to relieve internal stress. The [111] dimension was made short to minimize internal conical refraction effects. This geometry is also favorable for prestressing the sample in (111) [110] shear to introduce a large number of dislocations on the (111) plane with Burgers vector [110]. This biases the transformation to take place preferentially on one of the twelve slip systems. These dislocations may be excited in optical vibration by a (111) [112] ultrasonic stress wave. The yield stress was about $1.2 \times 10^8$ dynes/cm$^2$, and samples were strained by a few tenths of a percent.

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Velocity and decrement measurements were made using the highly sensitive pulse-echo superposition system developed by Holder and modified by Read and Holder. The system measures a change in superposition peak frequency related to the change in velocity by \( \Delta v = \frac{\Delta f}{f} + \frac{\Delta h}{h} \). Changes of 1 part in \( 10^7 \) are detectable if the temperature is suitably controlled.

Because the model makes predictions about the velocity change, and because the sample length changes by .39% during complete transformation, it is important to monitor the [111] dimension with a longitudinal strain gage. Typically it was found that in the temperature range of interest the length changes were relatively small so that \( (\Delta v/v) \times (\Delta f/f) \) within about 15%. Shear strain was also measured to see if the shear transformation strain was a function of prestressing shear strain.

Because cobalt-nickel is ferromagnetic there is a large background attenuation of the ultrasonic wave due to vibrating domain walls and eddy current damping. This can be greatly reduced by pinning the domain walls with a saturation magnetic field. Due to shape demagnetization it is easier to magnetize a sample along one of its long dimensions. Using the magnetization curves for Co_{65}Ni_{35} and calculating the demagnetization factor for an ellipsoid of our sample dimensions suggested that the external field needed to be 1.5 kilogauss along one of the long dimensions. (e.g., [110]). Measurements were made in an external field of 6.5 kilogauss.

Results of early runs were perplexing. Even though the samples had been pre-stressed to bias transformation on [111], the transformation lines were more prominent on (111), and (111) and absent on (111). Using small cube-shaped samples with magnetic field applied along different directions we found that the magnetic field was responsible for biasing transformation to occur on the sets of (111) planes most nearly perpendicular to the magnetic field direction. The reasons for this biasing are crystal anisotropy and magnetostriction. For our sample geometry the shape demagnetization factor along [111] would have required a field of 13 kilogauss for saturation, which was twice the field available. However, by putting chunks of Armco iron on the same lateral dimensions as our sample on either side, the sample was made to look more like a rod than a plate to the field so that magnetization to saturation along [111] could be achieved.

Co_{68}Ni_{32} has a transformation temperature \( M_s \sim -10^oC \). The temperature was ramped linearly down from room temperature at .1°C per minute. Since the effects we were looking for should occur at or immediately prior to the onset of transformation, for most runs the temperature was not allowed to go below \( M_s \) by more than a few degrees. If the transformation was allowed to proceed only this far it was found that by subsequently annealing the samples we could restore them to their initial state. By contrast, if the transformation was allowed to go to completion, near \(-140^oC\), the annealed sample exhibited a large decrease in \( M_s \) and even recrystallization in some cases.
3. **Results and Discussion.** — The sample designated $M_4$ was prestressed plastically in $(111) (110)$ shear by .7%. The superposition peak frequency is plotted versus temperature in Figure 1. The frequency is linear in temperature down to $-6^\circ C$ (normal background). Between $-6^\circ C$ and $-10^\circ C$ a dip in frequency with respect to the extrapolated line occurs. The dip is maximized near $-9^\circ C$ and begins to recover. Before recovery is complete however, just below $-10^\circ C$ the dramatic effects associated with transformation occur. The maximum fractional change at the dip is $1.7 \times 10^{-4}$.

Sample $M_5$ was cut from the section of ingot adjacent to $M_4$. It was prepared in the same way but was not prestressed after annealing. The superposition peak frequency is plotted versus temperature in Figure 2. The major difference between $M_4$ and $M_5$ is the absence of the dip immediately preceding transformation in $M_5$.

![Fig. 1](image1.png) **Fig. 1** — Pulse superposition frequency versus temperature for a $(111) (112)$ shear stress wave in sample $M4$ prestressed in shear to .7% strain.

![Fig. 2](image2.png) **Fig. 2** — Pulse superposition frequency versus temperature for a $(111) (112)$ shear stress wave in annealed sample $M5$. 

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To eliminate the possibility that the dip was due to an intrinsic difference between $M_4$ and $M_5$, the latter was annealed again and sheared to .2\% strain. Figure 3 shows the superposition peak frequency and attenuation versus temperature. A small dip in frequency is found with maximum fractional change being $5 \times 10^{-4}$. The rise in attenuation which occurs over the same temperature range as the dip is .075 db/\mu sec, or an increase in decrement of $8.2 \times 10^{-4}$.

The ratio of total decrement increase to the maximum fractional change in velocity is 16 which is greater than the $4\pi$ derived from the model. The discrepancy may be attributed to length changes and/or increased scattering near the transformation.

After a total of 21 ultrasonic measurement runs during the fcc-hcp transformation the following observations can be made:

a) The frequency dip in the optical mode occurred immediately prior to the transformation during the first cooling run after prestressing. If the sample was heated and cooled again, the dip was not observed.

b) The size of the dip was roughly proportional to the plastic prestrain:
\[
\frac{\Delta f_{\text{max}}}{f} \approx 0.025 \varepsilon.
\]

c) There was a scatter of more than $\pm 5\%$ in $M_5$ even for the first transformation of samples cut from the same ingot.

d) The largest shear strain observed was $5 \times 10^{-4}$ and the size and direction of strain was independent of plastic prestrain. The shear strain was, however, a useful indication of the onset of transformation.

e) It was shown to be possible to achieve nearly complete fcc single crystal to hcp single crystal transformation in a large sample by prestressing in [111] (110) shear and orienting a saturation magnetic field along [111].

The velocity and decrement in the vicinity of the dip are reminiscent of the model curves of Figure 2 of ref. 1. The magnitude of the dip in Figure 1 can be fit by assuming the density of extended dislocations responsible for the effect was $\Lambda = 2 \times 10^4 \text{cm}^{-2}$. The half maximum points of the dip occur near $-7.0^\circ$ and $-9.5^\circ$C. From Figure 2 of ref. 1, we find these points correspond to $\omega^{-1}_n = 3 \times 10^9 \text{sec}^{-1}$ and $1 \times 10^9 \text{sec}^{-1}$, or equilibrium widths of approximately $3 \times 10^{-5} \text{cm}$ and $10^{-4} \text{cm}$. Assuming the length of the extended dislocations between constrictions is $10^{-4} \text{cm}$, we find the stacking fault energy $\gamma$ at $-7.0^\circ$C and $-9.5^\circ$C is $-2.6 \text{ erg/cm}^2$ and $-2.9 \text{ erg/cm}^2$ respectively.
The values found above for the parameters are reasonable. The dislocation density is smaller than is typically found in fcc metals, but it is not the total dislocation density which is important here - only those largest dislocations on the verge of transformation. The chosen dislocation length is a reasonable choice for a long dislocation. Note too that the fault widths at the dip half maximum points are nearly as large as the assumed length as the instability is approached.

Due to the instability at transformation the entire dip was rarely traced out before the dramatic transformation effects occurred. The determining factor as to how much of the dip is seen is whether there are wide equilibrium spacings at which the dislocations can vibrate at low enough frequencies to be detected at 10 MHz. In the freshly annealed sample (Figure 2), there are relatively few dislocations and these are strongly pinned. Rather than quasi-statically breaking a few pins to assume successively wider spacings, the dislocations remain firmly pinned until catastrophically breaking away at the transformation.

In conclusion, our measurements in prestressed Co$_{68}$Ni$_{32}$ were consistent with the dislocation optical mode vibration model using reasonable values for the parameters of the model.

References
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