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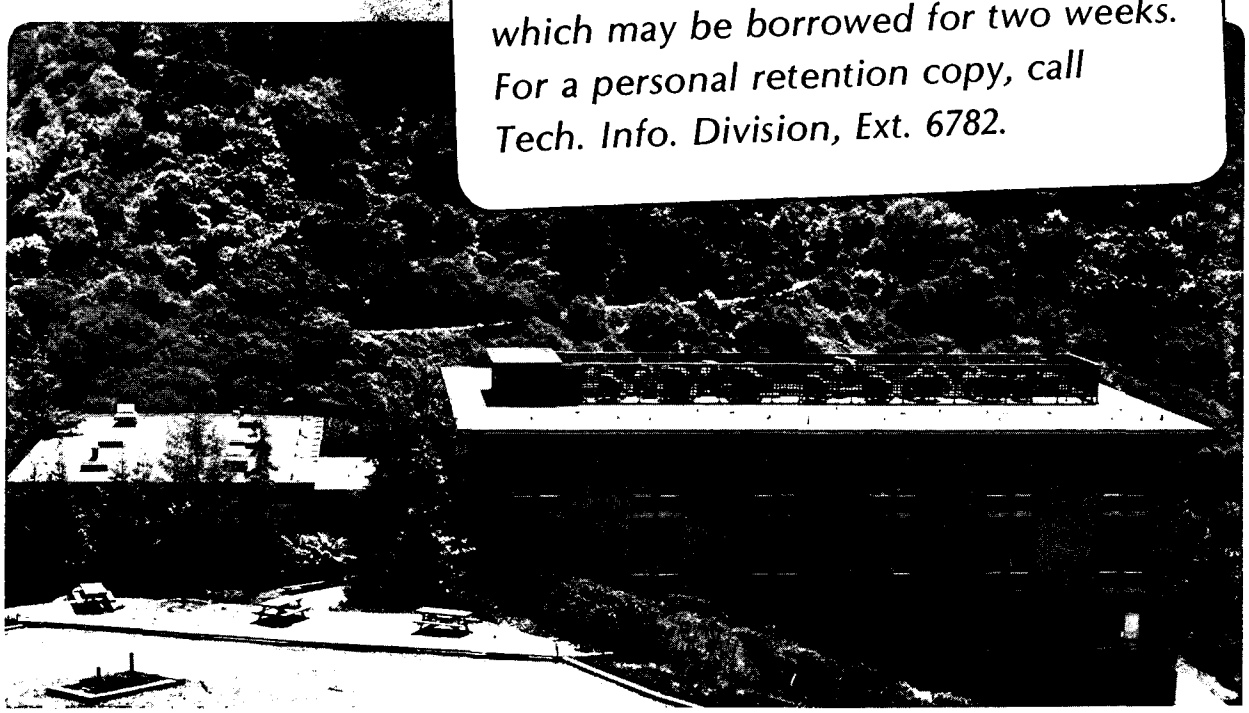

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THE EFFECTS OF HIGH MAGNETIC FIELDS ON THE MICROSTRUCTURE AND TOUGHNESS OF CRYOGENIC 9Ni STEEL

G. O. Fior, B. Fultz, and J. W. Morris, Jr.

Department of Materials Science and Mineral Engineering
University of California, Berkeley
and the
Materials and Molecular Research Division
Lawrence Berkeley Laboratory
Berkeley, California 94720
U.S.A.

Commercial 9Ni cryogenic steel was heat-treated to develop thermally unstable retained austenite. Some of this austenite was transformed to martensite by exposing the material to: 1) cryogenic temperatures, and 2) cryogenic temperatures plus 17 T magnetic fields. Those specimens exposed to the high magnetic fields consistently had the lower Charpy toughness. X-ray crystallography showed that the magnetic exposure caused additional $\gamma \rightarrow \alpha'$ transformation. Scanning electron fractography indicated that this additional $\gamma \rightarrow \alpha'$ transformation resulted in additional quasi-cleavage fracture, and therefore a lower toughness.

It is shown that the equilibrium thermodynamics of the coexistence of magnetic phases in magnetic fields cannot account for the large increases in the amount of $\gamma \rightarrow \alpha'$ transformation caused by the magnetic field. We therefore propose that our $\gamma \rightarrow \alpha'$ transformation is a process with an activation barrier that may be overcome by magnetic exposure. The effect of the $\gamma \rightarrow \alpha'$ transformation on the Charpy energy was mostly temperature-independent, however. This suggests that the mechanism of fracture associated with the $\gamma \rightarrow \alpha'$ transformation is not thermally activated.

1. Introduction

Fusion reactor machines employing magnetic confinement will require large superconducting magnets. Some materials used in the magnet structures will be simultaneously exposed to *large stresses, cryogenic temperatures,* and *high magnetic fields.* Ferritic steels offer some advantages to the cryogenic engineer who must design load-bearing components in superconducting magnets [1]. The high yield strength of ferritic steels is obtained economically. This high strength helps to minimize the amount of steel required in the magnet structure, further reducing the cost of the magnet and its cooldown. Ferritic steels also have a significant bulk magnetization, so their judicious placement may sometimes be used to increase the flux density of the magnet.

The often fatal limitation to the use of ferritic alloys at cryogenic temperatures is their ductile-to-brittle transition, which may result in an unacceptably low toughness at low temperatures. Adding Ni to ferritic alloys in even small amounts serves to suppress this ductile-to-brittle transition by preventing proeutectoid ferrite formation and by imparting more ductility to the martensite [2]. The microstructure of this material consists of laths of heavily dislocated martensite (α'-phase) which forms during the quench that terminates an austenitizing treatment. The ductile-to-brittle transition temperatures of 6Ni and 9Ni steels, however, are even more dramatically suppressed by a 1 hr intercritical tempering at 600°C, which produces a fine dispersion of austenite particles (γ-phase) of sub-micron dimensions situated between the martensite laths and on prior austenite grain boundaries [3,4]. A previous study indicated that this austenite does not transform to martensite upon cooling to 77 K, and is untransformed even after exposure to 16 T magnetic fields at 77 K [5]. The austenite stability may be reduced, however, if this material receives a different heat treatment. This may occur, for example, through a 20°C error in tempering temperature, or through welding or other heating of the magnet structure. In particular, the thermal stablity of austenite can be systematically reduced by increasing the tempering time. Materials with this thermally unstable austenite exhibit a reduced toughness at 290 K, and rapidly become brittle with decreasing temperature.

2. Effects of Magnetic Fields on Impact Toughness

In a previous study [5], overtempered 9Ni steel was exposed to 16 T magnetic pulses at 77 K. An additional transformation of austenite was observed in this material, along with a decrease in impact toughness at both 77 K and 290 K. The extra transformation of the austenite induced by the magnetic field served to reduce the impact toughness by nearly the same amount as did the thermal tansformation of the austenite (roughly 7 J/%γ), suggesting that exposure to magnetic fields affected the impact toughness solely by causing more $\gamma \rightarrow \alpha'$ transformation. However, these previous measurements were restricted to magnetic exposure at 77 K, so the effects of magnetic exposure and temperature were only crudely separated.

It is not obvious that the mechanical equivalence between thermally and magnetically transformed austenite found in the previous study holds true when the material is magnetically pulsed and broken at temperatures other than 77 K. The role of the austenite in the fracture process may change between 290 K and 77 K as the material passes through its ductile-to-brittle transition, and the mode of fracture changes from ductile-dimple to quasi-cleavage. Certain aspects of how temperature affects mechanisms of fracture may be explored by studying the effects of magnetic exposure at various temperatures. Specifically, if thermal activation is a controlling factor in those aspects of fracture associated with the $\gamma \rightarrow \alpha'$ transformation, then the mechanical consequences of the magnetically-induced $\gamma \rightarrow \alpha'$ transformation should be temperature-dependent. One goal of the present study was to increase, by magnetic exposure, the amount of the $\gamma \rightarrow \alpha'$ transformation in specimens cooled to temperatures in the range of the ductile-to-brittle transition. It should then be possible to isolate the temperature dependence of the relation between this extra $\gamma \rightarrow \alpha'$ transformation and the impact toughness.

3. Effects of Magnetic Fields on the $\gamma \rightarrow \alpha'$ Transformation

The second goal of the present study was to clarify the effects of temperature and magnetic field on the $\gamma \rightarrow \alpha'$ transformation itself. It is well-known that the free energy of the α'-phase becomes progressively less than that of the γ-phase as the temperature is reduced. At every temperature, however, the magnetic field will modify the total thermodynamic potentials, Φ_α and Φ_γ, of the α'-phase and the γ-phase from their usual Gibbs free energy, G, at zero magnetic field:

$$\Phi_{\alpha'} = G_{\alpha'} - M_{\alpha'} \cdot H \ , \quad \Phi_\gamma = G_\gamma - M_\gamma \cdot H \ , \qquad 1)$$

where H is the applied magnetic field, and M is the magnetization of the phase. In analogy to the Clausius-Clapeyron derivation of the slope of phase boundaries in equilibrium T-P phase diagrams [6], the following slope of the γ-α' phase boundary in a T-H phase diagram is obtained [7]:

$$\frac{dT}{dH} = \frac{M_\alpha(T) - M_\gamma(T)}{S_\gamma(T) - S_\alpha(T)} \ . \qquad 2)$$

The difference $M_\alpha - M_\gamma$ is known to be about 2 μ_B/atom . Knowing the entropy difference, $S_\gamma(T) - S_\alpha(T)$, is equivalent to knowing the latent heat of the martensite transformation, which is simply this entropy difference times the temperature of the phase transformation. Both S and M are only weakly dependent on T, so eqn 2 predicts a constant "temperature equivalence" for the strength of the magnetic field in shifting the temperature of γ-α' equilibrium.

The martensite transformation is known to exhibit many non-equilibrium characteristics. Therefore, in addition to their equilibrium roles of affecting the $\gamma \rightarrow \alpha'$ transformation through the T·S and M·H terms in Φ, we should also expect that the intensive variables T and H may affect kinetic factors such as nucleation of the $\gamma \rightarrow \alpha'$ transformation. It was hoped that this work would help clarify whether the $\gamma \rightarrow \alpha'$ transformation is aided by thermal activation, or perhaps even an effective "magnetic activation". From eqn 2 we expect that the characteristic curves of the volume fraction of austenite, f_γ, versus T will be horizontally shifted to larger T with the application of high magnetic fields. The amount of shift should approximate the shift found in the previous Soviet work [7,8]. Any large departure from this behavior will indicate an effect of either temperature or magnetic field on the kinetics of the $\gamma \rightarrow \alpha'$ transformation.

4. Experimental

Cryogenic 9Ni steel was kindly supplied by the Nippon Kokan Company with the commercial QT heat treatment. Plates of this material were given the following heat treatment to produce unstable austenite: (1000°C 3 hrs /WQ), (800°C 1.5 hrs /WQ), (600°C 300 hrs /WQ). For comparison of thermal and thermo-magnetic effects, matched pairs of Charpy bars were cut from equivalent sections of these plates.

A magnetic pulse with a peak field of 17 T was developed for about 10 msec by discharging a capacitor bank into a copper solenoid immersed in liquid nitrogen. Virtually all of the $\gamma \rightarrow \alpha'$ transformation occurred after two such pulses, so a sequence of 5 pulses was used for the magnetic exposure. The eddy current heating of the specimens was measured, and was found to be less than 2 K. The specimens were isolated from the liquid nitrogen bath of the solenoid by an evacuated anti-cryostat. The temperature of the specimens was controlled to better than ±5 K by a Lake Shore Cryotronics DRC-70C controller with a Si diode temperature sensor in the feedback loop. The power to the electrical resistance heater surrounding the Charpy bar was up to 35 watts, depending on temperature and the gas pressure in the anti-cryostat. To ensure meaningful comparison of the two specimens in a matched pair, the specimen that was not exposed to the magnetic field was also cooled in an identical manner with the same apparatus. Before fracture, the temperature of the Charpy specimens was obtained by reducing the temperature of an isopentane bath with liquid nitrogen.

A Picker Nuclear x-ray diffractometer with Cu Kα radiation was used for measuring the amount of austenite in the specimens at room temperature before and after each thermal or thermo-magnetic treatment. The Charpy specimens were deeply polished on their back sides for phase analysis, and three orientations of the specimen in the diffractometer were used for each datum. The diffraction peaks were digitized, and the method of R. L. Miller was used to quantify the volume fraction of austenite [10]. The fractographic study employed a JEOL JSM-U3 scanning electron microscope operated at 15kV with secon-

dary electron detection. Areal fractions of quasi-cleavage and ductile-dimple fracture were determined by digitizing the area of each type of fracture in two-dimensional fractographs. Five fractographs were obtained from each specimen.

5. Results and Discussion

Magnetic Effects on the $\gamma \rightarrow \alpha'$ Transformation

Previous measurements at 77 K [5] of the effect of high magnetic fields on the amount of transformation of retained austenite in overtempered 9Ni steel were confirmed (1.3% in the previous study versus 1.7% here). However, in the present study twice that amount of austenite was observed to have been magnetically transformed in specimens exposed to the magnetic fields around 160 K. We note that this is approximately the temperature at which the equilibrium thermodynamic theory of phase stability in magnetic fields predicts the largest effects. However, the equilibrium effect of the magnetic field should be to shift the zero-field curve of fig 1 rigidly upwards in temperature by the result of eqn 2 times H. We can estimate this horizontal shift of the zero-field curve by appeal to previous Soviet work, which was claimed to be consistent with the equilibrium thermodynamic theory [7,8,10]. The composition of our austenite is probably similar to the Soviet Fe-18Ni-0.4C alloy 50Kh2N22, so our net entropy difference, $S_\gamma - S_{\alpha'}$, will also be similar. Our magnetization difference is similar to that in the Soviet study [8], and since our $\gamma \rightarrow \alpha'$ transformation occurs at only a slightly higher temperature than theirs, we expect a ~ 30 K horizontal shift of our zero-field curve in fig 1. This expected shift is indicated by the dashed curve. We see that this predicted shift is less than one-third as large as the horizontal shift of the two experimental curves. The equilibrium thermodynamic model is incapable of accounting for our large observed shift. Furthermore, at temperatures below 200 K there is an unexpected upward shift of the $\Delta f\gamma$ versus T curve in addition to the horizontal shift.

It appears that either temperature or magnetic field helps to overcome some sort of a nonequilibrium activation barrier in the transformation from γ-phase to α'-phase. That there exists an activation barrier to the $\gamma \rightarrow \alpha'$ transformation is not surprising; the transformation in this material is extremely athermal*, and the difference between M_s and M_b is hundreds of °C. A thermal activation explanation of the magnetic shift of the $\Delta f\gamma$ vs. T curve could be consistent with fig 1 if the $\gamma \rightarrow \alpha'$ transformation at low temperatures ($\lesssim$ 50 K) has trouble surmounting the activation barrier, and the magnetic exposure is thus unimportant. However, although our present data only extend to 77 K,

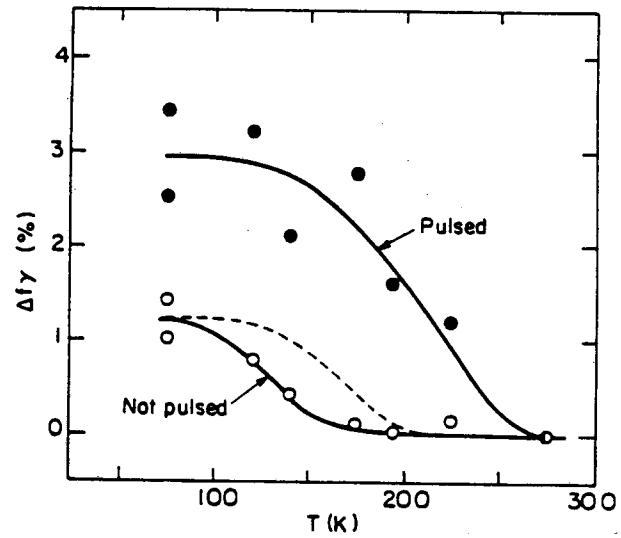

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Fig. 1 Reduction in $f\gamma$ with respect to $f\gamma$ at room temperature immediately after tempering. Measured values of $f\gamma$ at room temperature immediately after tempering varied from 12 to 17%. Solid points represent specimens which received five 17 T magnetic pulses at the temperature indicated, open points represent specimens which were exposed to the low temperature only.

it appears that the magnetic exosure at low temperatures is important. Furthermore, at higher temperatures the $\gamma \rightarrow \alpha'$ transformation should not be limited so much by thermal activation as by the need for more thermodynamic driving force. However, since very large effects (i.e. much greater than the thermodynamically predicted ~ 30 K shift) are seen in fig 1 at the higher temperatures ($\gtrsim$150 K), this thermal activation hypothesis does not seem to be supported by our data.

An explanation that invokes a non-equilibrium "magnetic activation" is perhaps more consistent with fig 1, especially if we later find that the amount of $\gamma \rightarrow \alpha'$ transformation at temperatures below 77 K reaches a plateau. At lower temperatures magnetic activation should result in an upward displacement of the zero-field $\Delta f\gamma$ versus T curve of fig 1. The temperature-dependence of this upward shift should depend on the distribution of austenite stabilities, and possibly on the need for thermal activation. At higher temperatures it is possible that magnetic activation could initiate a $\gamma \rightarrow \alpha'$ transformation where none occurred under thermal exposure alone. Within these wide theoretical bounds, a form of "magnetic activation" is consistent with the magnetic effect seen in fig 1.

Unfortunately, a plausible mechanism for magnetic activation remains elusive. It could be possible that the magnetic activation mechanism involves internal stresses that are developed during the magnetic exposure. Such stresses may serve to aid the nucleation of the martensite transformation.

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* In a previous study of this same material [11], the amounts of $\gamma \rightarrow \alpha'$ transformation after 10^{-1}sec, 10^{2}sec, and 10^{8}sec were found to be the same.

Pulsed magnetic fields generate transient eddy currents near the surfaces of electrically conducting materials. Lorentz forces between these currents and the applied field will squeeze the material. Our estimates of this effect show that the maximum plausible stress is less than 10% of the yield stress. In a Soviet study the strengths of these eddy currents induced by pulsed fields were varied by electroplating some of their specimens so that they would have different surface electrical resistivities [10]. This work showed that the stresses due to eddy currents were unimportant in inducing further martensite transformation. We measured the heating due to eddy currents in our specimens, and found it to be only ~ 2 K, which was even less than that found in the Soviet work. Forced magnetostriction has been suggested [8] as a possible cause of some of the magnetically-induced $\gamma \rightarrow \alpha'$ transformation. However, it appears to us that these magnetostrictive stresses will also be much less than the yield stress. Although the transformation of the austenite in 9Ni steel with the commercial QT heat treatment can only be induced by plastic strain, we have not yet checked to see if the transformation of the austenite in our overtempered 9Ni steel can be induced by low stresses at cryogenic temperatures. Also, allowances for local stress concentrations could perhaps make magnetostrictive stresses and eddy current induced stresses plausible mechanisms of magnetic activation. We should also leave open the possibility of some form of synergetic thermo-magnetic activation, at least until the scaling of $\Delta f\gamma$ with H is explored.

Magnetic Effects on the Charpy Toughness

The specimens in each matched pair of Charpy specimens were broken at low temperatures after thermal exposure, and after thermal plus magnetic exposure. The results are shown in fig 2. Although we did not intend to compare systematically materials with different amounts of austenite, there was some initial variation in the amount of austenite present in our heat treated material. This variation may be caused by differences in the quench that terminated the tempering treatment; specimens receiving a more rapid quench had less austenite than those that were quenched less effectively. The initial variation in the amount of austenite at room temperature is responsible for some variations in the absolute amount of Cvn in fig 2, but the difference between specimens in each matched pair was quite consistent. The correlation between the total amount of austenite in a specimen and its Charpy impact energy, shown in fig 3, is not so convincing as the correlation of fig 4. Fig 4 shows that the transformation of the austenite in overtempered 9Ni steel reduces the Charpy energy by more than 5 J/%γ when the $\gamma \rightarrow \alpha'$ transformation is either thermally or thermomagnetically induced. This agrees with the

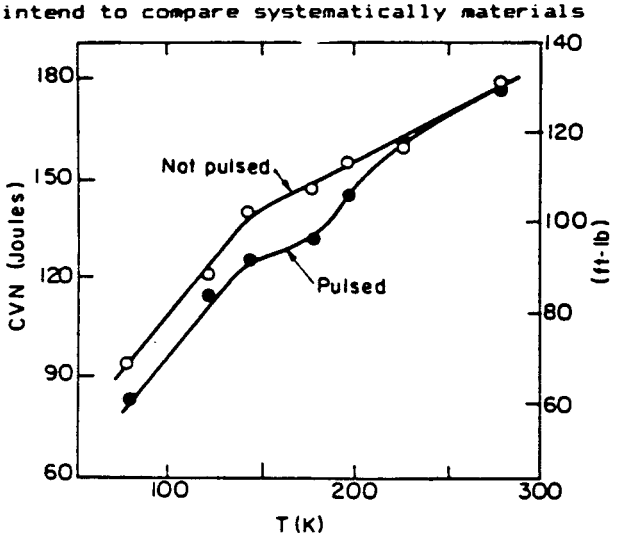

Fig. 2 Charpy impact energy of specimens broken at the indicated temperatures. Solid points and open points represent the same specimens used for the data of fig 1.

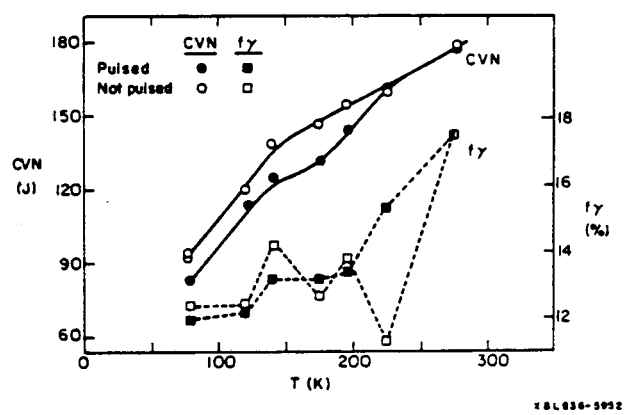

Fig. 3 Charpy energy of fig 2 shown with the total amount of austenite measured just prior to impact testing.

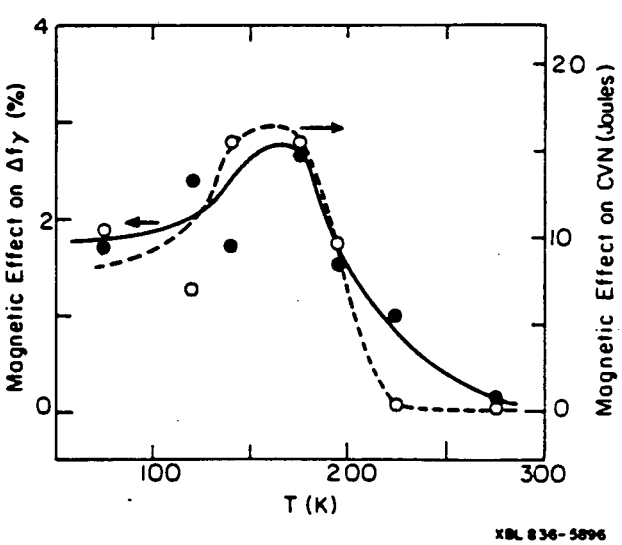

Fig. 4 Difference of the curves shown in figs 1 and 2). Solid points: magnetic effect on the volume fraction of austenite. Open points: magnetic effect on Charpy energy.

results of our previous study [5], which reported an effect of nearly 7 J/%γ. The data of these two figures suggest that the presence of austenite in this material is beneficial to Charpy toughness primarily because its transformation to martensite is detrimental. Assuming that all the specimens developed the same amount of austenite during the tempering treatment, the transformation of the austenite upon cooling to room temperature must be less detrimental to mechanical properties than the transformation of the austenite at temperatures below room temperature.

Our SEM fractography study showed that the magnetically-induced decreases in Charpy energy found in the matched pairs of specimens were associated with an increase in the amount of quasi-cleavage fracture at the expense of ductile-dimple fracture. A 1% increase in areal fraction of quasi-cleavage fracture corresponded to a decrease in Charpy energy of about 1.5J. This is in excellent agreement with a previous study [12] of an embrittled 6Ni steel broken at temperatures in the range of its ductile-to-brittle transition. This previous study found that a decrease in Charpy energy of 1.6J corresponded to a 1% increase in quasi-cleavage fracture, but we believe the precision of this agreement to be largely coincidental. Since the increase in quasi-cleavage fracture correlates with the reduction in Charpy energy, and the amount of γ→α′ transformation also correlates with the reduction in Charpy energy, clearly the increase in the amount of quasi-cleavage fracture correlates with the amount of γ→α′ transformation. If we hypothesize a causal relation in which the γ→α′ transformation forces an increase in the amount of quasi-cleavage fracture, a detailed picture of the effect of magnetic fields on the Charpy energy can be drawn.

In this picture the magnetic field causes additional γ→α′ transformation through equilibrium and non-equilibrium thermodynamic effects. Material with this extra γ→α′ transformation has a microstructure that is more prone to quasi-cleavage fracture, so when it is later fractured it exhibits a reduced Charpy impact energy. We believe that the effect of the γ→α′ transformation on the amount of quasi-cleavage fracture is more convincingly shown in the present study than in a simple temperature-dependent fractographic study; other properties of the material that relate to fracture may be temperature-dependent in addition to the amount of γ→α′ transformation. The fact that the magnetically-induced and thermally-induced γ→α′ transformations produce the same mechanical effect is convincing evidence that there is a causal relationship between the amount of γ→α′ transformation and the amount of quasi-cleavage fracture.

Fig 4 shows that the relation between the amount of magnetically-induced γ→α′ transformation and the reduction in Charpy energy is not temperature-dependent. Because a mechanism requiring thermal activation would reduce the effect of the extra γ→α′ transformation at low temperatures, this indicates that the mechanism through which the γ→α′ transformation affects the fracture mode is probably not thermally activated over the temperature range 77 K - 290 K. Two important implications follow. First, thermal activation apparently plays an unimportant role in the microstructural changes induced by the γ→α′ transformation. Second, it appears that thermal activation also plays an unimportant role in the fracture mechanism affected by these microstructural changes. Since it is the process of quasi-cleavage fracture that is apparently promoted by the additional γ→α′ transformation, this second implication indicates that quasi-cleavage fracture is not a thermally activated process.

Further studies of magneto-mechanical phenomena are necessary before their engineering importance can be assessed. Cryogenic 9Ni steel was given a heat treatment which would hopefully be avoided in practice. Even with its unstable austenite, fig 2 shows that the effect of 17 T magnetic fields on the ductile-to-brittle transition is not large. However, we believe that the situation could be different if the magnetic exposure occurred *during* fracture.

7. Conclusions

The previously reported γ→α′ transformation and the corresponding reduction in Charpy energy induced in overtempered 9Ni steel by high magnetic fields at low temperatures were confirmed. The amount of magnetically-induced γ→α′ transformation is far greater than expected from considerations of equilibrium thermodynamics. We propose that this additional γ→α′ transformation occurs by a mechanism which has a large activation barrier. Our present data suggest that this barrier is overcome more effectively by "magnetic activation" than by thermal activation.

The magnetically-induced reductions in Charpy energy are associated with an increase in the areal fraction of quasi-cleavage fracture at the expense of ductile-dimple fracture. We propose that the additional γ→α′ transformation induced by magnetic exposure is responsible for additional quasi-cleavage fracture, and a consequent reduction in Charpy energy. The effect of a given amount of γ→α′ transformation in reducing the Charpy energy is not temperature-dependent. This suggests that the mechanism of magnetically-induced quasi-cleavage fracture is not thermally activated.

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