CONSEQUENCES OF THE RE-TRANSFORMATION OF PRECIPITATED ADSTENITE IN FERRITIC CRYOGENIC STEELS

https://escholarship.org/uc/item/9s12d4xr

Morris Jr., J.W.

2010-02-12

Peer reviewed
Presented at the International Conference on Martensitic Transformations, Massachusetts Institute of Technology, Cambridge, MA, June 24-29, 1979

CONSEQUENCES OF THE RE-TRANSFORMATION OF PRECIPITATED AUSTENITE IN FERRITIC CRYOGENIC STEELS

J. W. Morris, Jr., C. K. Syn, J. I. Kim, and B. Fultz

June 1979

Prepared for the U.S. Department of Energy under Contract W-7405-ENG-48
CONSEQUENCES OF THE RE-TRANSFORMATION OF PRECIPITATED AUSTENITE IN FERRITIC CRYOGENIC STEELS

By

J. W. Morris, Jr., C. K. Syn, J. I. Kim, and B. Fultz

Department of Materials Science and Engineering
University of California, Berkeley

Technical Report No. 8
Office of Naval Research
Contract No. N00014-75-6-0154
The re-transformation of precipitated austenite ($\gamma_\text{p}$) in Fe-(6-9)$_2$Ni steels has been investigated, with particular emphasis on the influence of transformation characteristics on alloy toughness at cryogenic temperature. In all cases studied the precipitated austenite re-transforms, either during cooling to cryogenic temperature or during deformation prior to fracture of the alloy. When the re-transformation is thermally induced the product martensite tends to form in a variant identical to that present before precipitation of the austenite; no significant refinement of the microstructure is achieved. On the other hand, mechanical transformation of the austenite produces a distribution of martensite variants determined by the local strain, hence preserving a refined microstructure conducive to low temperature toughness.

I. Introduction

Ferritic Fe-Ni steels which are intended for structural use at cryogenic temperatures are given a final intercritical temper, i.e., a temper in the two-phase ($\alpha+\gamma$) region of the phase diagram, which has the effect of precipitating austenite phase along the lath boundaries and prior austenite grain boundaries of the parent martensite. The intercritical temper substantially lowers the ductile-brittle transition temperature of the alloy, often by as much as 200°C. This improvement is generally attributed to the retention of a small fraction of precipitated austenite in the product microstructure. However, the mechanism of toughening remains unclear.

Previous research on the influence of retained austenite on the toughness of cryogenic steels suggests two qualitative sources for the decrease in the ductile-brittle transition temperature. First, the austenite will getter deleterious species, such as carbon, which segregate to the austenite during the precipitation. Second, the austenite may serve to blunt sharp-fronted cracks in the martensite, preventing them from causing catastrophic failure.

The "gettering" action of precipitated austenite is experimentally verified and certainly contributes to alloy toughness. But gettering by itself cannot fully account for the beneficial influence of retained austenite. The introduction of a small fraction of precipitated austenite...
austenite also decreases the ductile-brittle transition temperature of Fe-Ni alloys which are nominally interstitial-free and chemically get-tered. (8) We have, therefore, undertaken research to clarify the "crack-blunting" action of retained austenite. The results of this research suggest that the "crack-blunting" effect is intimately connected to the eventual martensitic transformation of the retained austenite and is particularly sensitive to the orientation of the product phase.

II. Thermomechanical Stability of Precipitated Austenite

Research on the mechanical behavior of intercritically tempered cryogenic steels gives a consistent indication that the beneficial influence of retained austenite is related to its thermodynamic stability. (1-3) The stability of the precipitated austenite decreases if the tempering temperature is too high, which presumably causes the austenite to become relatively lean in stabilizing solutes, or if the tempering time is too long, which leads to an increase in the size of the austenite particles. Both excessive tempering temperature and excessive tempering time cause a rise in the ductile-brittle transition temperature.

The sensitivity of alloy toughness to austenite stability could be easily explained if the austenite were mechanically as well as thermally stable at test temperature, since the relative ductility of austenite islands would serve to blunt propagating fractures. However, recent research conclusively demonstrates that the retained austenite present in tough cryogenic steels is mechanically unstable, and undergoes an essentially complete transformation to martensite prior to fracture.

The mechanical instability of precipitated austenite was first shown by Kim and Schwartz (9) and by Fultz (10) who used Mossbauer spectroscopy to analyse the residual phase distribution near fracture surfaces in cryogenic steels, and found an essentially complete transformation of retained austenite during ductile fracture of both 6Ni and 9Ni steels at cryogenic temperature. These studies were extended by Syn, et al. (11) who prepared profile fractographic specimens and conducted transmission electron microscopic studies of the deformed material immediately adjacent to ductile fracture surfaces in 9Ni steel (Fig. 1). They confirmed the mechanical transformation of retained austenite and showed that the transformation product is a dislocated martensite, whose presence in the matrix is not incompatible with high toughness. More recent research on samples pulled in tension at 77°k (Fig. 2) revealed that the transformation of austenite in tough cryogenic steels is strain-induced and completed at relatively modest levels of plastic strain.

The demonstration that the austenite retained in tough cryogenic steels is fully transformed at strains well below those associated with fracture raises a puzzle as to why it is necessary (as it appears to be) that the austenite be present in the first place. The puzzle is compounded by the results of transmission electron microscopic studies (Fig. 3) which show that the austenite which spontaneously transforms on cooling, even near 77°k, yields a dislocated martensite which is apparently identical in substructure to that induced by plastic strain. The resolution of the puzzle appears to lie in a qualitative difference in the orientations of the two kinds of martensite with respect to the
parent matrix, an orientational difference which has its source in the pervasive memory of thermally-induced martensitic transformations in Fe-Ni steels.

III. The Role of Precipitated Austenite in Microstructure Refinement

On quenching, Fe-(5-9)Ni cryogenic steels develop a microstructure which is made up of laths of dislocated martensite which tend to be organized into packets. These packets may be 10-20μm in size and consist of bundles of laths which bear a close crystallographic relation to one another. While the laths themselves may be small (<1μm in diameter) their close alignment in packets has the consequence that the salient mechanical properties of the alloy tend to be controlled by the packet size rather than by the smaller lath size.

The influence of packet alignment on fracture is illustrated in Fig. 4, which shows a profile scanning electron fractograph of a brittle fracture surface in 6Ni cryogenic steel. Long, flat cleavage traces are apparent; these traverse martensite packets. Crystallographic analysis shows that the facets follow (100) cleavage planes which are nearly common to the packet. Where the packet is not well defined the fracture surface is rough and often locally ductile. Observations such as these suggest that a major goal of the processing of cryogenic steels should be to destroy long-range packet alignment.

An intercritical temper superficially appears to offer an effective means for decomposing aligned packets. During the temper austenite islands nucleate along the lath boundaries and essentially isolate the laths from one another. There is, however, a subtle memory in the austenite precipitation and its reversion to martensite on cooling which vitiates packet decomposition. As illustrated in Figs. 3 and 5 the austenite precipitated within a given packet has a strong statistical tendency to form in a single variant, which is K-S related to the martensite matrix, and, if it re-transforms on cooling, to regenerate the martensite which gave it birth. If the result of an intercritical temper is to precipitate a thermally unstable austenite only slight refinement of the microstructure is accomplished and no significant improvement in mechanical properties should be anticipated. In fact, the transition temperature often rises after such a temper.(12)

If, on the other hand, the intercritical temper leads to the retention of a reasonable distribution of interlath austenite then this austenite remains until it is transformed by plastic strain. Initial studies of mechanically transformed austenite, exemplified by the micrograph shown in Fig. 4, suggest that the martensite variant formed in this case is that most compatible with the local strain, hence tending to be the variant most compatible with the nominal strain of the sample. The product martensite is K-S related to its parent austenite, which is in turn K-S related to the matrix; however, the K-S relationship permits a sufficient number of variants that the martensite will only rarely share a well-oriented cleavage plane with the matrix.

The precipitation of thermally stable austenite hence achieves a decomposition of the packet alignment of martensite laths which is largely retained even if the austenite transforms mechanically prior to fracture.
A substantial decrease in the ductile-brittle transition temperature of the alloy is therefore expected and is usually observed.

IV. Conclusion

The precipitation of austenite during an intercritical temper of Fe-(6-9)Ni martensite will decrease the ductile-brittle transition temperature of the alloy if it breaks up the packet alignment of martensite laths. Because of memory effects in the re-transformation of the precipitated austenite it is necessary that the austenite have sufficient thermal stability for a significant fraction of it to be retained on cooling to the test temperature. Subsequent mechanically-induced transformation of the austenite does not then eliminate its beneficial effect.

Acknowledgment

This research was supported by the Office of Naval Research under Contract No. N00014-75-6-0154 and by the Division of Materials Sciences, Office of Basic Energy Sciences, U. S. Department of Energy under Contract No. W-7405-Eng-48.

References

Fig. 1 Profile TEM fractograph and diffraction pattern in 9Ni steel. The fracture surface (indicated with arrows) was plated with iron (above the boundary indicated with arrows) to prepare thin foils of the fracture surface layer. The diffraction pattern was taken from the central region right below the boundary line. The circled area shows γ-like particles which have transformed to α'.

Fig. 2 Martensite Transformation of the termally stable and unstable γ in 9Ni steel during tensile tests at 77°K. Remaining fraction of γ with respect to the original amount at room temperature was plotted against the tensile strain ε.

Fig. 3 TEM of the thermally unstable γ in 9Ni steel. The γ has transformed to highly dislocated α' on quenching to 77°K from room temperature. It has transformed to the same orientation of the matrix.

Fig. 5 Typical microstructure of the thermally stable γ at 77°K in 9Ni steel. γ particles are K-S related to the matrix of tempered α'.
Fig. 4 Profile SEM fractograph showing the lath orientations with respect to the fracture crack path in 6Ni steel. Arrows indicate regions where the crack deviates.

Fig. 6 Partial transformation of γ to α' in 9Ni steel during tensile test at 77°K. The dark field pictures show the γ and tensile strain-induced α' from the γ.
The re-transformation of precipitated austenite ($\gamma_p$) in Fe-$(6-9)$%Ni steels has been investigated, with particular emphasis on the influence of transformation characteristics on alloy toughness at cryogenic temperature. In all cases studied the precipitated austenite re-transforms either during cooling to cryogenic temperature or during deformation prior to fracture of the alloy. When the re-transformation is thermally induced, the product martensite tends to form in a variant identical to that present before re-precipitation of the austenite; no significant refinement of the microstructure.
is achieved. On the other hand, mechanical transformation of the austenite produces a distribution of martensite variants determined by the local strain, hence preserving a refined microstructure conducive to low temperature toughness.