The Influence of Martensite and Ferrite on the Properties of Two-Phase Stainless Steels Having Microduplex Structures

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The mechanical properties of a series of stainless steels ranging in composition from 16.5 pct Cr, 5.5 pct Ni to 23.9 pct Cr, 2.9 pct Ni have been determined. The series of alloys lie along an approximate 1700°F tie line with room temperature microstructures ranging from 100 pct martensite to 100 pct ferrite. Yield and tensile strengths increased directly with increasing martensite content. In alloys containing on the order of 40 to 60 pct martensite, the presence of a fine dispersion of tougher, albeit stronger, martensite was quite effective in lowering the ductile-to-brittle impact transition temperature.

RECENT studies of two-phase Fe-Ni-Cr alloys have shown that many compositions having a suitably fine (microduplex) grain size exhibit the phenomenon of superplasticity at temperatures similar to and in excess of approximately one-half the absolute melting temperature.[1,2] In addition to this unusual high temperature behavior, many of these alloys have useful intermediate and low temperature properties. In particular, studies of two-phase iron-base compositions have led to the development of the stainless steel IN-744 having a composition of 26 pct Cr, 6.5 pct Ni.[2] This alloy has an excellent combination of the mechanical properties coupled with very good corrosion resistance.

The present authors have reported studies of the deformation and fracture characteristics of IN-744.[3] The tensile deformation characteristics seemed closely akin to the behavior of ferritic stainless steel. However, the two-phase alloy had a ductile-to-brittle Charpy V-notch transition on the order of 300°F lower than that for a ferritic stainless steel having the composition of the ferrite of the two-phase alloy (approximately 32 pct Cr-3 pct Ni). In a subsequent publication,[4] the mechanical behavior of a series of alloys lying along a tie-line from 32 pct Cr-3 pct Ni-balance Fe to 19.5 pct Cr-9 pct Ni-balance Fe were investigated. It was found that very useful combinations of strength and toughness could be obtained in alloys with microduplex structures containing approximately 40 to 50 pct austenite. The improved toughness of the two phase alloy was related to two factors: 1) the greater difficulty of cleavage crack initiation afforded by the grain refinement of the ferrite phase which is easily attained in two-phase alloys, and 2) blunting of propagating cleavage cracks by softer, tougher, and transformable (to martensite) austenite particles.

In a later investigation,[5] an alloy of 50 pct Fe-50 pct Cu having a microduplex structure was analyzed. Again it was found that the ductile-to-brittle transition temperature was about 300°F lower than that of the tie-line iron phase. The improvement in toughness was relatable to the difficulty of cleavage crack initiation and to crack blunting by softer, tougher, but nontransformable copper particles. This study demonstrated that metastability of the second phase is not a necessary condition for toughening in microduplex alloys.

The present investigation was undertaken to determine if similar toughening behavior could be attained in stainless steels having a microduplex structure composed of a soft, cleavage-prone ferrite phase and a stronger, tougher phase, in this case martensite.

EXPERIMENTAL PROCEDURE

All of the present alloys were prepared as 30 lb vacuum induction melts from virgin raw materials. The compositions of the present series of "tie-line" alloys are listed in Table I. The percent ferrite in each heat, determined by lineal analysis, is included in the table. The "tie-line" was estimated by consideration of several factors: 1) microprobe analysis of a much richer two-phase alloy composition annealed at 1700°F,[6] 2) the tie-lines at 1832°F determined by Pilliar and Kirkaldy,[7] and 3) phase analyses of nominal 21 pct Cr, 5 pct Ni, 0.3 pct Ti steels by Sergeeva and Khaklova.[8] Fig. 1 compares the "tie-line" of the present work with that discussed in Ref. 4.

All the heats were cast as 4 by 4 in. ingots. The ingots were soaked at 2200°F and rolled to 2 by 2 in. billets. These billets were subsequently rolled to $\frac{3}{4}$ in. diam bar stock from 1700°F.

$\frac{1}{4}$ in. diam, $1\frac{1}{4}$ in. gage length tensile specimens, and standard Charpy V-notch impact specimens were machined from the as-rolled bar. After machining, the specimens were annealed in argon for 1 hr at 1700°F followed by either air cooling or water quenching.

Table I. Chemical Composition of Materials Investigated

Heat	Ni	Cr	C	Al	Ti	Pct α
1	5.54	16.5	0.007	0.05	0.18	8.4
2	5.37	17.2	0.007	0.06	0.16	15.6
3	4.69	18.8	0.022	0.06	0.16	39.5
4	4.18	20.2	0.011	0.05	0.16	62.9
5	3 54	21.9	0.017	0.06	0.16	87.3
6	2.85	23.9	0.022	0.06	0.16	100

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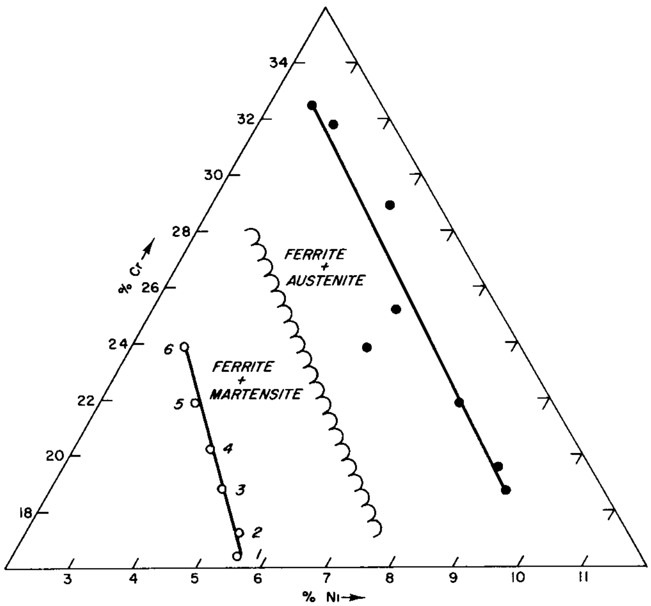


Fig. 1—Ternary diagram of materials used in this investigation as well as those discussed in Ref. 4. Closed circles indicate alloys studied in Ref. 4.

All samples were then refrigerated 16 hr at −106°F to ensure complete transformation of the austenite to martensite.

The resultant microstructures were very similar in character to those found in the austenite-ferrite alloys.[4] Alloys in the central portion of the two-phase region showed ferrite matrices containing elongated fibers of martensite. The grain sizes of both phases were quite small, on the order of several microns. The alloys at the ends of the tie-lines were generally equiaxed in character and the grain sizes were approximately 25 μ.

The tensile specimens were tested at temperatures between room temperature and −320°F, using appropriate liquid equilibrating baths for the low temperature tests. Charpy specimens were tested at temperatures ranging from −320° to 212°F.

RESULTS AND DISCUSSION

Tensile Behavior

The yield and tensile strengths of the heats were determined at various temperatures after annealing 1 hr at 1700°F and water quenching. These results are shown in Fig. 2. It can be seen that there is a reasonably linear relation between yield strength and percent ferrite at all test temperatures. This might be expected inasmuch as the two components of the ferrite-martensite heats have bcc structures and similar work hardening behavior. The variations in grain size evidently had little effect on the tensile properties.

The variation of room temperature yield strength with percent ferrite of both the ferrite-austenite heats of Ref. 4 and ferrite-martensite heats, all annealed 1 hr 1700°F and either water quenched or air cooled, is shown in Fig. 3. In the case of the ferrite-austenite series, composed of bcc and fcc phases, the observed nonlinear change of yield strength with ferrite content is probably related to the different work hardening behavior of the two phases.[4] As had been observed in the ferrite-austenite tie-line, the ferrite-martensite series also shows that with higher ferrite contents air cooling led to higher yield strengths than water quenching.

In summary, the tensile properties of the ferrite-martensite seem to be consistent with the entirely bcc structures of the alloys. In contrast to the ferrite-austenite alloys of Ref. 4, increasing ferrite contents lead to lower, rather than higher yield strengths. In both series of alloys, ultimate tensile strengths decreased with increasing ferrite content.

Fracture Behavior

The Charpy V-notch impact energies of the tie-line series of heats are shown as a function of test temperature in Fig. 4. Samples were annealed 1 hr at 1700°F and either air cooled or water quenched, as noted. As in the ferrite-austenite tie-line, the slower cooling rate significantly raised the ductile-brittle transition temperature of the more ferritic alloys. There was very little effect of cooling rate on the impact properties of the more martensitic alloys.

The impact results are replotted in Fig. 5 as impact energy vs ferrite content for several different test temperatures. Selecting an impact energy of 40 ft-lb as a transition from ductile to brittle behavior, the

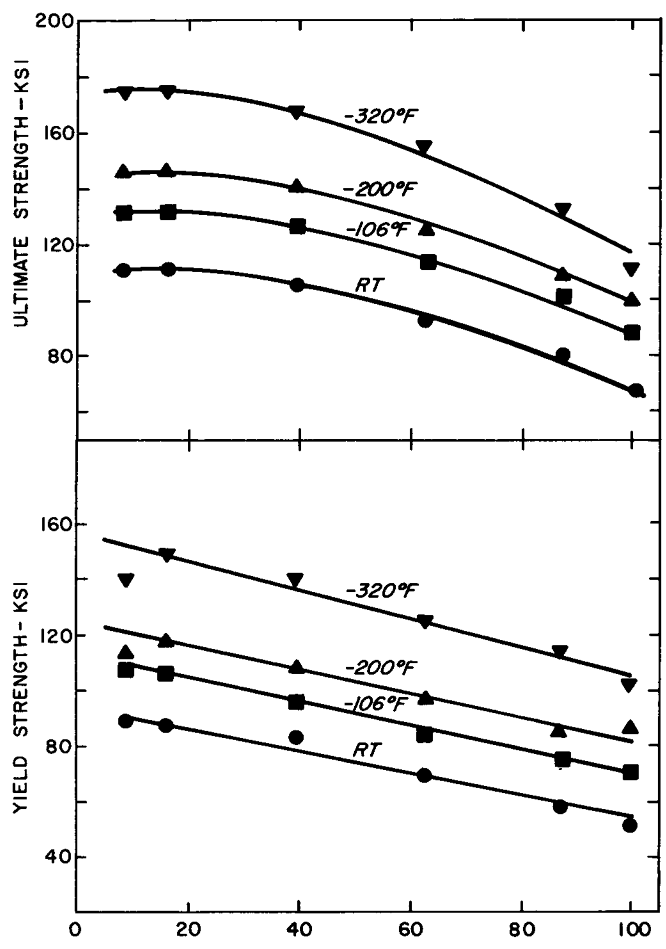


Fig. 2—0.2 pct offset yield strength and ultimate tensile strengths of ferrite-martensite alloys vs ferrite content.

transition temperature for both air cooled and water quenched samples is shown as a function of ferrite content in Fig. 6. In going from alloys which are all ferritic to alloys having on the order of 40 to 50 pct martensite, it can be seen that there was a lowering of the ductile-to-brittle impact transition temperature on the order of 500°F for air cooled samples and 300°F for water quenched samples. These results are similar to results observed in microduplex structures composed of ferrite and austenite[4] and iron and copper.[5] In distinction to the two above mentioned studies, the present results indicate that a cleavage-prone phase (ferrite) can be toughened by a fine dispersion of a stronger, tougher phase (martensite) as well as by a dispersion of a weaker, tougher phase (austenite or copper).

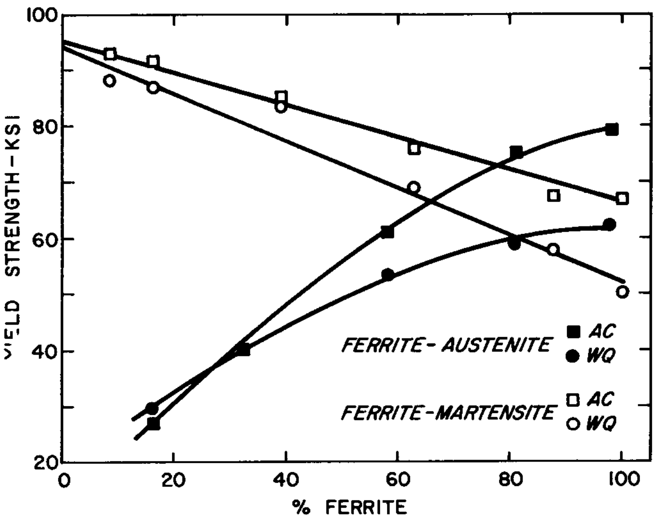
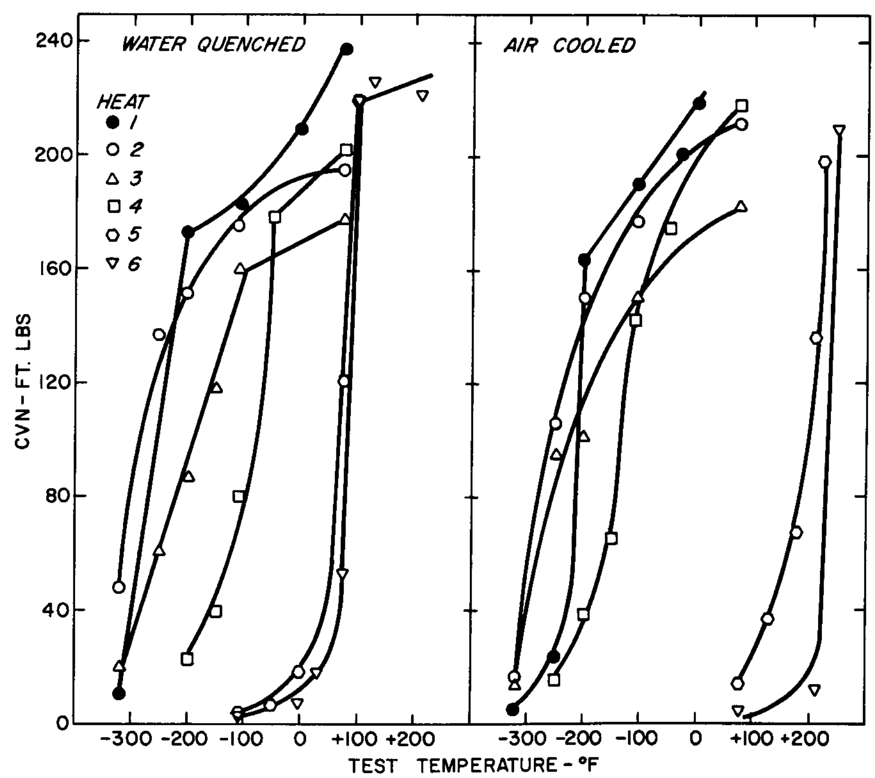


Fig. 3—Room temperature 0.2 pct yield strengths of ferrite-martensite and ferrite-austenite, Ref. 4, alloys vs ferrite content.

In order to determine if the toughening effects of finely dispersed martensite derived from its influence on crack initiation or crack propagation, standard Charpy V-notch samples of all heats were annealed 1 hr 1700°F, water quenched, and then fatigue cracked to a depth of about 0.031 in. below the machined notch before testing. The introduction of a sharper notch would be expected to lower the crack initiation energy, but not affect the crack propagation behavior. The results are shown in Fig. 7. Impact transition temperatures were determined by selecting an energy of 400 ft-lb per sq in. as a ductile-to-brittle transition. This is approximately equivalent to 40 ft-lb for standard Charpy specimens. These are shown with the transition temperatures of standard Charpy samples in Fig. 6. In going from the completely ferritic alloy to alloys containing 40 to 60 pct martensite the transition temperature was lowered on the order of 250°F. The results indicate that when the energy expended in crack initiation was lowered by fatigue precracking, the transition temperatures of highly ferritic alloys were increased by about only 70°F while the transition temperatures of highly martensitic alloys were increased on the order of 140°F.

Fig. 8 shows results of impact tests on fatigue precracked and standard Charpy samples of alloys containing approximately 8, 40, and 100 pct ferrite plotted in terms of impact energy per unit area vs test temperature. The difference between the two curves for each alloy should be a measure of the change in crack initiation energy. At subzero temperatures there was obviously little difference in energy absorbed by the ferrite, whereas the 92 pct martensite alloy showed significant differences over the whole range of temperature. The 40 pct ferrite alloy was similar to the 92

Fig. 4—Charpy V-notch impact energies of air cooled and water quenched samples as a function of test temperature.



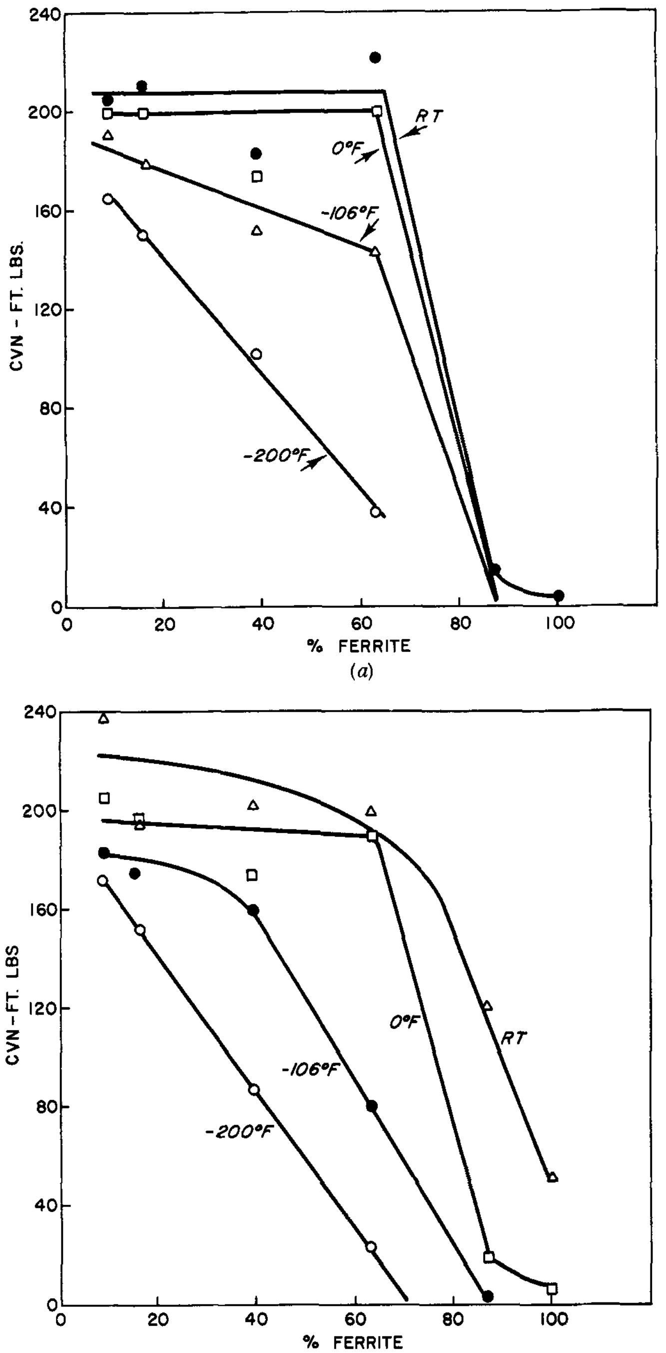


Fig. 5—Charpy V-notch impact energy as a function of ferrite content (*a*) air cooled, (*b*) water quenched.

pct martensite alloy, showing distinct differences in energies over the whole temperature range.

These results indicate that the presence of martensite in the microstructure significantly raised the crack initiation energy of the two-phase alloys. This effect could have resulted from either the suppression of cleavage initiation in the ferrite, or to an effect of the martensite in minimizing premature propagation of any cleavage cracks that might have formed in the ferrite.

As a further means of studying the effects of microstructure on fracture initiation, a series of double V-notched tensile samples were tested at −200°F. The rationale for these tests was that during loading both notches would deform in similar fashion and that crack initiation might occur in both notches prior to fast crack propagation from one of the notches. The unbroken notch was then sectioned and examined metallographically to reveal what had happened prior to fast crack propagation. The specimens showed increasing crack opening displacements with increased martensite contents. In alloys containing on the order of more than 40 pct martensite, however, there was little or no evidence of microcracking in the ferrite phase. Thus, the presence of a fine martensite distribution significantly retarded crack initiation in the ferrite.

One reason for this effect of martensite is a rather indirect one, namely the much finer grain size of the ferrite when martensite is present. The reduction in grain size is a result of the retardation of grain

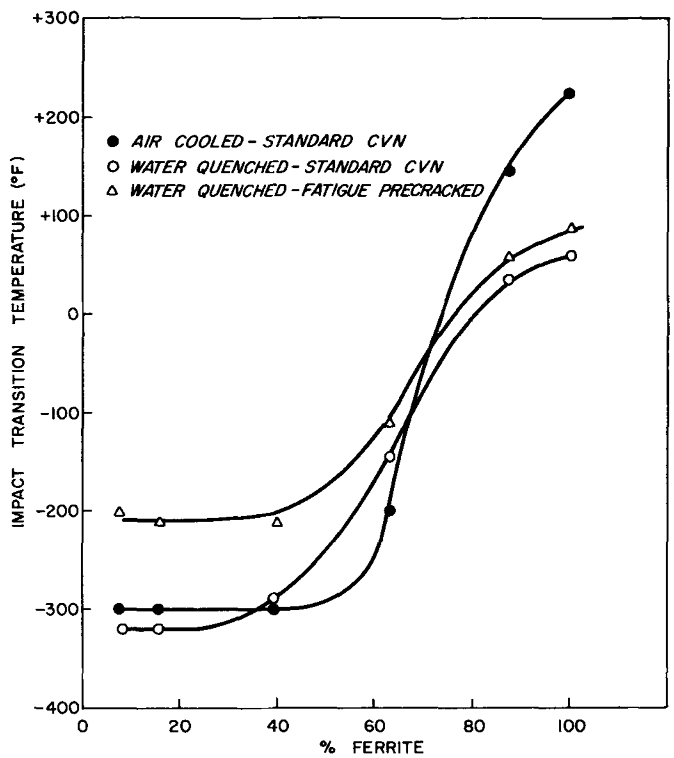


Fig. 6—40 ft-lb impact transition temperature as a function of ferrite content.

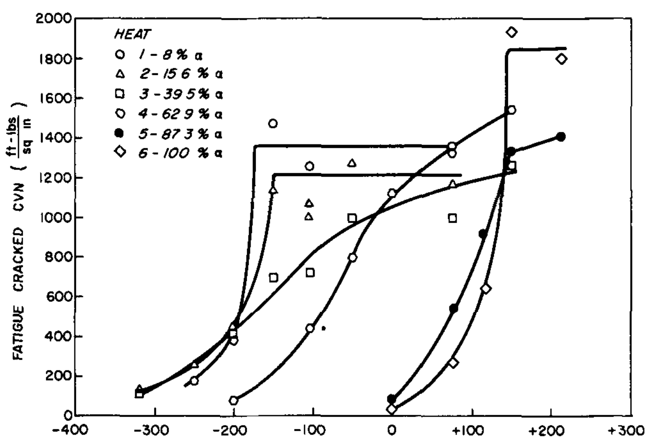


Fig. 7—Impact energy per unit area of fatigue precracked CVN samples vs ferrite content.

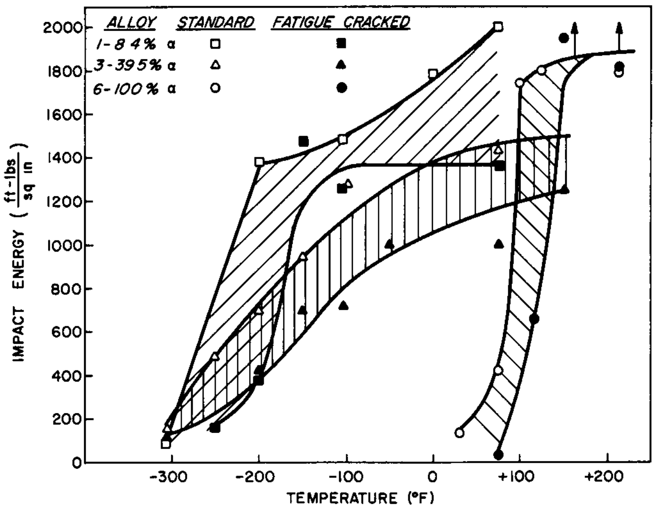


Fig. 8—Impact energy per unit area of three representative alloys for both standard and fatigue precracked CVN samples vs test temperature.

growth during hot working or annealing when large amounts of two phases are present. Thus, in any microduplex alloy some improvement in toughness might be obtained because of a reduction in grain size. It should be pointed out however, that in both the present alloys and also the austenite-ferrite alloys the ferrite grain size was comparably small in most of the two-phase alloys in the tie lines, but the toughness was continuously improved by increasing amounts of the tougher phase. Therefore the reduction in ferrite grain size is not the sole reason for the better toughness.

A second factor that may influence crack initiation in the ferrite is stress relaxation by plastic flow in the tougher phase. In the austenite-ferrite and Fe-Cu alloys the tougher phases had significantly lower yield strengths than the ferrites. In the present alloys the 0.2 pct offset yield strength of the martensite was higher than the ferrite, but the elastic limit was considerably lower. Thus, in all three systems the ability of the tougher phase to deform at relatively low stresses could help relieve local stresses in the ferrite that would otherwise initiate cleavage.

A number of the fractured tensile and impact specimens were nickel plated on the fracture surfaces. The crack propagation behavior was then studied by cross sectioning and metallographically examining the samples. As expected at low temperatures failure of the ferrite was almost entirely by cleavage, whereas the martensite failed primarily by a ductile mode except at the lowest temperatures where some cleavage was observed. Occasional delamination fractures perpendicular to the main cracks were observed but these did not appear to be a significant factor. Numerous instances of cleavage cracks in the ferrite that were stopped when they reached a martensite layer were observed. An example is shown in Fig. 9. This same type of crack blunting was also commonly observed in the other microduplex alloys. It seems quite likely, as in the other alloys, that the ability to stop or at least delay cleavage propagation in this manner must significantly raise the crack propagation energy and make an important contribution to the improvement in toughness.

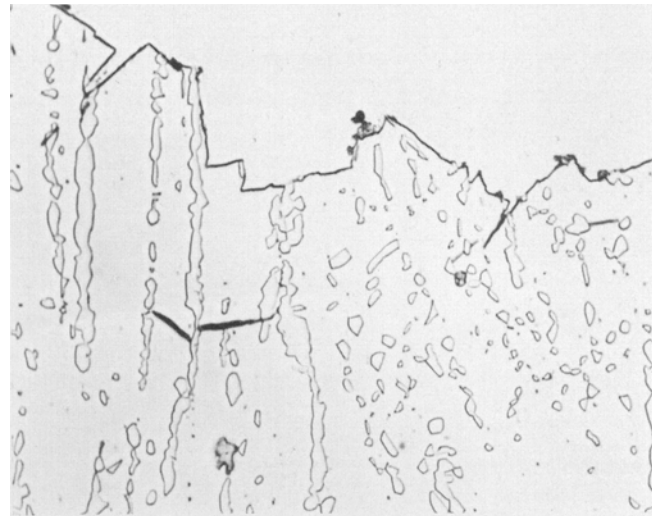


Fig. 9—Example of cleavage cracks in the ferrite, phase being stopped and blunted by martensite particles in a Charpy sample of Heat 5 tested at −200°F. 10 pct oxalic acid etch. Magnification 690 times.

CONCLUSIONS

1) The presence of a strong tough martensite phase in microduplex ferrite-martensite stainless steels leads to improved strength and toughness over the level of properties of a completely ferritic alloy.

2) At roughly 50 pct martensite, the impact transition temperature is significantly lower than that of an all-ferritic alloy. This result is similar to results for microduplex ferrite-austenite stainless steels and microduplex Fe-Cu alloys.

3) The present results suggest that the primary prerequisite for a second phase to toughen a cleavage prone matrix is that the second phase be tougher than the primary phase. Strength differences between the two phases or transformability of the second phase do not seem to be significant for producing lower transition temperatures.

4) The improved toughness of microduplex alloys appears to be related to the effects of a finely dispersed tough second phase on both the energies for crack initiation and crack propagation.

5) From a practical point of view the best combination of strength and toughness over the ferrite-martensite tie-line series is obtained for an all-martensitic alloy, rather than at a 50-50 phase ratio which had been observed for the more useful ferrite-austenite stainless steels.

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