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THE EFFECT OF MICROSTRUCTURE ON THE MECHANICAL PROPERTIES OF A COMMERCIAL 12Cr-1Mo STEEL (HT-9)

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The microstructure of a commercial 12Cr-1Mo steel (HT-9) and its associated effect on strength and toughness properties is being studied in a continuing program aimed at qualifying the alloy for use in fusion energy machines. Interim results show this alloy is subject to a degree of tempered martensite embrittlement and temper embrittlement. For applications projected for fusion machines at lower temperatures, a new heat treatment (1000C, 1 hr, air-cooled followed by 650C tempering) at lower temperatures and shorter times than the vendor-recommended heat treatment has been identified. Microstructural differences between the treatments are discussed, and mechanical properties are correlated.

1. INTRODUCTION

The class of steels containing 12Cr-1Mo has been used extensively in fossil-fueled power generation plants in Europe for two decades(1,2). These materials contain enough chromium to give corrosion resistance; however, the total alloy content is limited to insure that the material can be heated to a fully austenitic phase field and subsequently transformed to martensite by air-cooling(3). The condition in which it has been used is sufficiently overtempered (e.g., 2-1/2 hr at 760C) to insure microstructural stability for long service at 600C. The microstructure in this state is still martensitic, in that the lath structure is retained, although the dislocation structure has recovered sufficiently such that it contains ferrite and the laths are surrounded by carbides(1). The presence of secondary carbide forming elements such as chromium, tungsten, molybdenum, and vanadium causes alloy carbides to precipitate and, combined with a very fine lath structure, gives this material superior creep resistance(5,6). The U.S. fusion effort is interested in a 12Cr-1Mo-0.3V-0.5W steel (HT-9) because it has shown much less swelling due to irradiation than austenitic steels(7). The neutronic environment in fusion machines will be significantly more energetic than that for fast reactors from which these data were generated. It is therefore expected that results for 12Cr-1Mo at higher fluence levels will demonstrate its superiority more dramatically. The next generation fusion machine, called the Fusion Engineering Device (FED), has low lifetime fluences and thus will not utilize 12Cr-1Mo alloy except for material test panels. However, this alloy has been designated as the reference material for the first wall/blankets in several future fusion machines(8,9). Due to the critical temperature control required for martensitic steels in fabrication and welding, the Alloy Development for Irradiation Performance (ADIP) task group from the Office of Fusion Energy has been studying these and other aspects to determine the feasibility of 12Cr-1Mo for use in the sort of structures envisioned for tokamaks. An understanding of the basic metallurgy of this specific alloy is critical in determining the microstructural relationships to the observed mechanical properties in the fabricated, heat-treated, and welded conditions and relating them to postirradiation properties and microstructures. It is also noted that the vendor heat treatment has been developed for applications at 600C. In most designs, fusion first walls are at lower temperatures, and it is suggested that heat treatment variations may yield a fundamentally superior microstructure for resistance to irradiation damage and thermal embrittlement.

This paper presents interim results of a continuing program to determine the effect of thermal aging of 12Cr-1Mo-0.3V-0.5W steel (HT-9) on several microstructures produced by different heat treatments, and comparisons of the results with those using the vendor-recommended heat treatment.

2. EXPERIMENTAL

The chemistry of the steel is Fe-0.21C-1.01Mo-0.54W-0.33V-0.58Ni-0.50Mn-0.21Si-0.008P-0.003Si. The reader is referred to Ref. 10 for all the necessary experimental details.

3. RESULTS AND DISCUSSION

Figure 1 shows the effect of austenization temperature on the hardness and prior austenitic grain size of the 12Cr-1Mo steel held for one hr at temperature and air-cooled. Grain sizes were determined by comparison at 100X to a standard template. If the material during austenitization is fully in the fcc-austenite phase field, the as-quenched hardness is a function of the carbon content of the steel(11,12). Therefore, the hardness is a relative measure of the carbide dissolution at different temperatures. This can be seen in Fig. 1. From 950 to 1100C, the hardness increases from R_c 41 and remains nearly constant above 1000C at R_c 46-47. From 900 to 950C the hardness increases signifi-

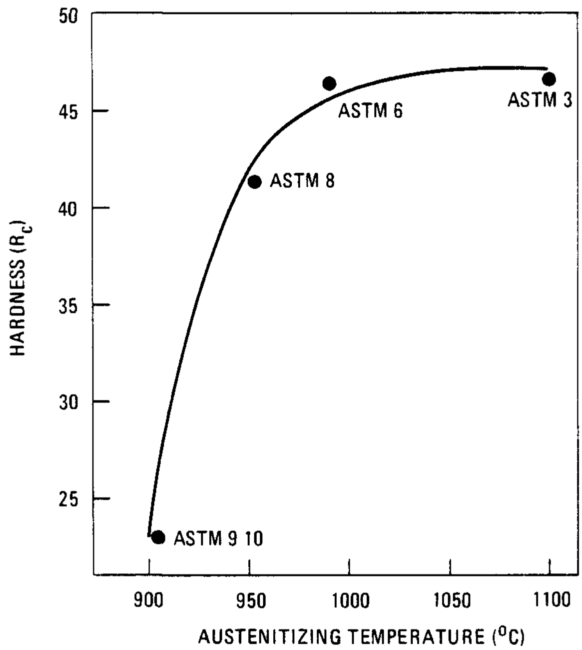

Figure 1 The effect of austenitization temperature on the hardness and grain size of 12Cr-1Mo (HT-9) steel heated for an hour and air cooled.

cantly. Optical metallography showed that this was related to the presence of ferrite at the lower temperature. The carbides which remained undissolved from 950 to 1100C were extracted and determined to be $M_{23}C_6$ with a lattice parameter of 1.064 nm. The amount extracted decreased as the austenitization temperature increased. The effect of tempering temperature on hardness as a function of austenitizing temperature is shown in Figure 2. There are several features to note. First, this material displays the characteristic secondary hardening profile. The strong carbide-forming elements Cr, Mo, W, and V will precipitate as secondary carbides (that is, after the primary M_3C has overaged and dissolved) on dislocations which serves to pin them. It is this pinning action which increases the yield stress. Secondly, the peak hardness occurs around 500C and the peak height is greatest at higher austenitizing temperatures. This is an important indication of how prior microstructure (e.g., undissolved carbides) can influence mechanical properties. More carbon and secondary hardening elements are available for precipitation due to dissolving $M_{23}C_6$. Austenitizing at 1000 and 1050C yields the same secondary hardening peak hardness, indicating the $M_{23}C_6$ carbide was relatively stable over that range for 1 hr. However, austenitizing at 1100C yields a higher secondary peak, R_c 51, up from R_c 47. This is probably due to the strong influence of small increases in carbon content of the matrix as $M_{23}C_6$ dissolves(13). Finally, the overaging characteristics appear different for higher austenitization temperature. Fig. 2 shows that strength decreases more rapidly as a function of tempering temperature when the austenitization is at higher temperatures.

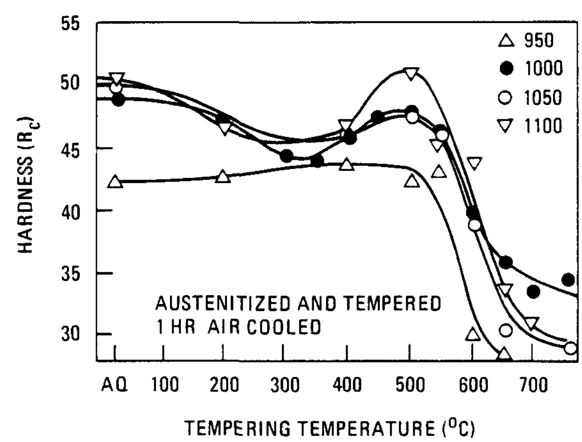

Figure 2 The effect of tempering temperature on hardness as a function of austenitization temperature.

The effect of tempering on the room temperature Charpy impact toughness is shown in Fig. 3. Dynamic yield stress is reported along with the hardness (which is a function of the ultimate tensile stress), and these are compared to impact toughness. The toughness values increase from the as-quenched to 200C tempered condition. At 250 to 350C it levels off at 20J even though hardness was decreasing through this range, but increases to 30J at 400C. This is probably due to tempered martensite embrittlement (also called '350C' or '500F' or 'one-step temper'(14) embrittlement). The large decrease in toughness from 450 and to 550C is in the temper embrittlement regime for this material(15) and is probably due to tramp impurity elements such as S and P causing a 'micropollution'(16) of boundaries, rendering them weaker. More work is being done to determine the exact cause. At higher temperatures the material loses strength sufficiently to allow much more yielding prior to failure, which is manifested by an increase in toughness.

4. CORRELATION OF MICROSTRUCTURE AND MECHANICAL PROPERTIES

This section discusses continuing microstructural observations. Figure 4 shows a transmission electron micrograph of the 1000C as-quenched condition. It is typical of interlocking lath martensite structures. An arrow points to an undissolved $M_{23}C_6$ carbide. Dark field diffraction analysis in part (b) showed that some austenite was retained at the martensite lath boundaries as thin films, approximately 200A

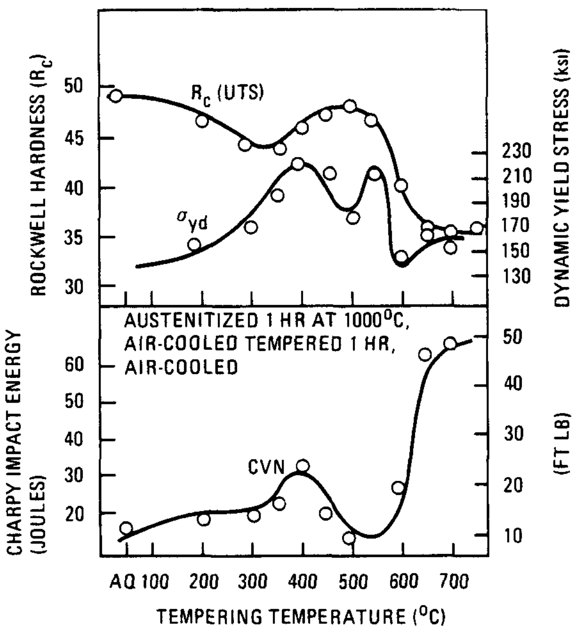

Figure 3 The effect of tempering temperature on hardness, dynamic yield stress, and Charpy impact toughness after austenitizing 1 hr at 1000C and tempering for 1 hr, both followed by air-cooling.

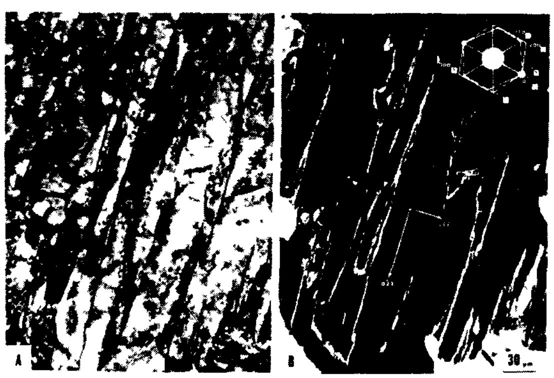

Figure 4 Transmission electron micrograph of 1000C, 1 hr, air-cooled microstructure showing (a) interlocking dislocated lath martensite, and (b) a dark field showing thin films of retained austenite surrounding the laths.

thick. This has been reported previously and is the cause of subsequent tempered martensite embrittlement when the austenite decomposes(17). The austenitized and air-cooled from 1050C condition was also observed. There was an increase in the amount of retained austenite at the lath boundaries. This also has been reported for a silicon-modified 4340 steel(18) and a 300-M steel(19). Tempering will subsequently cause the retained austenite films to decompose. Observations show this is complete by 350C. In Fig. 3 there was a pause in the expected increase in toughness values between 250 and 350C. These carbides are of the M_3C type. At 500C carbides have precipitated within the martensite laths causing the secondary hardening peak. These have the morphology of M_2C but as yet are not identified. Results from similar studies have reported Mo_2C as the strengthening carbide(20), although that is known to compete with MC carbides in some cases(21). The structure after austenitizing at 1000C and tempering at 650C is shown in Fig. 5. The martensite laths are surrounded by $M_{23}C_6$ carbides forming a semicontinuous network. In this condition the impact energy has increased significantly to ~70J, indicative of a loss in strength. The microstructure shows that all precipitation is now in the lath boundary. Because carbides no longer pin dislocations, the dynamic yield stress decreases in this condition, as seen in Fig. 3.

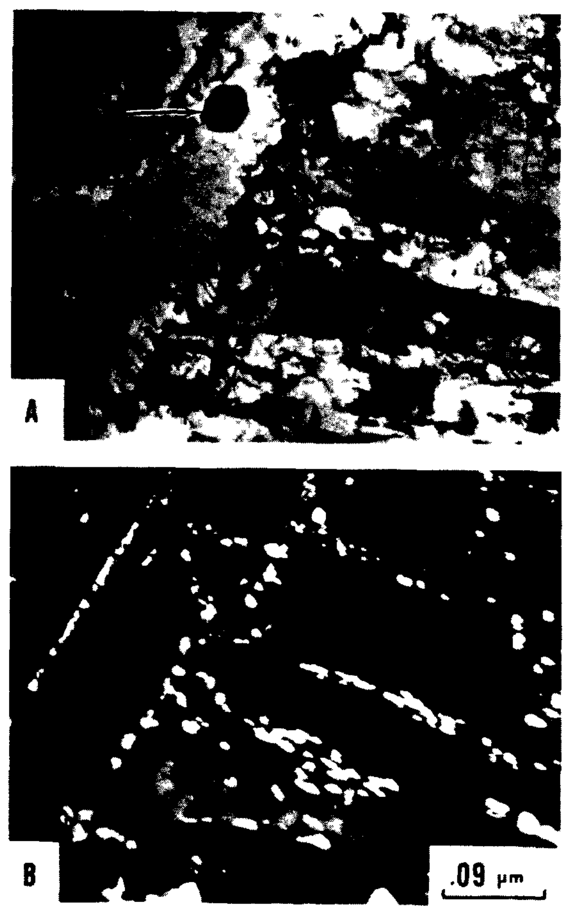

Figure 5 Transmission electron micrograph of quenched and tempered condition (1000C, 650C, both 1 hr, air-cooled) showing that the retained austenite films decompose to $M_{23}C_6$ carbides. This has been called tempered martensite embrittlement.

The 12Cr-1Mo steels exhibit microstructural stability. Recently it was shown that the martensite lath structure remains intact even after 80,000 hr at 600C(2), although there is significant subgrain formation. The microstructure of the 12Cr steel after austenitizing and tempering at 1000 and 650C for 1 hr and air-cooling, and subsequently aged at 550C for 100 hr was similar to that shown in Fig. 5. There was little more precipitation of $M_{23}C_6$ carbide due to thermal aging, indicating the quenched and tempered condition to be close to equilibrium. This was also true for the lath and carbide morphology. Precracked and instrumented Charpy impact test results are shown in Fig. 6. It can be seen that the ductile-brittle transition temperature (DBTT) has increased 60C. The fracture mode from the upper-shelf knee and lower temperatures was interlath cleavage with some ductile tearing between laths in all cases. Concurrent with this, standard Charpy tests were performed. The results did not indicate an increase in the DBTT, that is the blunt notch test was insensitive to this degree of embrittlement.

5. SUMMARY

From the data presented several conclusions can be drawn which relate the microstructural relationships to mechanical properties. It has been established that the 12Cr-1Mo steel (HT-9) is subject to tempered martensite embrittlement, which is a loss in toughness when the material is tempered near 350C. This is due to retained austenite films around martensite laths decomposing to M_3C. Furthermore, even at higher tempering temperatures, the films decompose and leave semicontinuous networks of $M_{23}C_6$ carbides at the lath boundaries. These may act as crack nucleators, and the fracture surfaces appear to have cleaved at the martensite lath boundaries. Also, temper embrittlement was observed in that there was a shift in DBTT when the material was aged at 550C for 100 hr while there was no discernible change in microstructure.

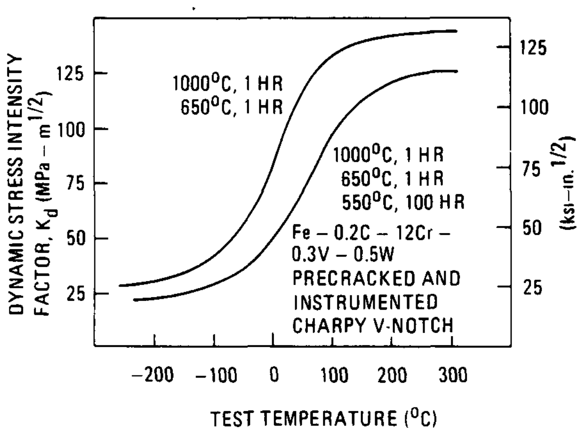

Figure 6 The effect of test temperature on the precracked Charpy impact toughness, K_d, after aging for 100 hr at 550C. All fracture modes were interlath cleavage with some ductile tearing between them.

The fracture data suggest that blunt notched tests are less sensitive measurements of embrittlement. A realistic flaw in a structure would be very sharp, such as a fatigue crack; therefore, it is suggested that a true picture of the degree of embrittlement is obtained from measurement of fatigue cracked specimens. Also the small size of many irradiation specimens if precracked will yield more constraint and therefore a truer measure of embrittlement.

6. ACKNOWLEDGEMENTS

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