The Influence of Intercritical Heat Treatment on the Temper Embrittlement Susceptibility of a P-Doped Ni-Cr Steel

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Temper embrittlement of a Ni-Cr steel doped with 0.06 wt pct P aged at 480°C after an intercritical heat treatment (IHT) has been compared with that of the conventionally heat treated steel with a range of prior austenite grain sizes. The IHT virtually eliminated the embrittlement susceptibility, but low temperature brittle fracture was still intergranular. It appears that most of the benefit of IHT in this steel comes from microstructural refinement; however, IHT also reduced the amount of segregation of Ni and P to grain boundaries. This is believed to be connected with partitioning of Cr during IHT.

THE susceptibility of alloy steels to temper embrittlement can be reduced significantly by what is known as an ‘intercritical’ heat treatment. Instead of the conventional austenitization above the Ac3 temperature, the steel is quenched from below this temperature, so that its microstructure prior to quenching comprises a mixture of the γ and α phases. A lower-than-usual tempering temperature is necessary to obtain a hardness equivalent to that of the conventionally treated steel.

The reason for the reduced embrittlement susceptibility is still a matter for conjecture. In addition to the microstructural refinement, which is known to reduce embrittlement susceptibility, it is possible that benefits accrue from solute partitioning between the α and γ phases, and perhaps from the formation of carbides during the intercritical treatment. Also, there is some question about the stability of the resistance to embrittlement in components which must operate for long times at elevated temperatures.

In an attempt to elucidate the mechanism of the effect of IHT, a study was made of temper embrittlement induced by P and Sb in a simple Ni-Cr steel which has been extensively studied and characterized. The approach was simply to compare the embrittlement of intercritically heat treated (IHT) steel and conventionally heat treated (CHT) steel, both with the same hardness, taking account of the effect of grain size variation in the CHT steel. This paper reports the work on the P-doped steel; the Sb-doped steel will be reported upon subsequently.

EXPERIMENTAL PROCEDURE

The alloy, whose composition is given in Table I, was vacuum-induction melted from electrolytic iron and high purity alloy additions, cast in an argon atmosphere, and then forged and hot swaged to 6.35 mm diam rod.

The heat treatments employed are described schematically in Fig. 1. Austenitization was carried out in a dynamic vacuum of <10^-2 Torr; tempering and aging were done in evacuated capsules. The three austenitizing temperatures produced austenite grain sizes of ASTM No. 4, 6 and 9, respectively. The Ac3 temperature of this steel was found to be 767 ± 2°C by dilatometry and hardness measurements on quenched samples, and an IHT temperature of 745°C was selected on this basis. Previous work by Ohtani on a similar steel had indicated the Ac1 temperature to be around 675°C. It was found that a tempering treatment of 2 h at 560°C in the IHT specimens gave the same hardness as 1 h at 625°C in the CHT.

Table I. Alloy Composition

<table>
<thead>
<tr>
<th>C</th>
<th>Ni</th>
<th>Cr</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4</td>
<td>3.5</td>
<td>1.7</td>
<td>0.061</td>
</tr>
</tbody>
</table>

Fig. 1—Schematic representation of heat treatments.
Diamond pyramid hardness (DPH) measurements were made using a 1 kg load.

Ductile-brittle transition measurements were made, as in previous work,\(^4,5,7,8\) by cantilever bending tests on circumferentially notched 5.84 mm diam rods in an Instron machine using a deflection rate of 5 mm/min. The area under the load-displacement curve, as recorded on a strip chart, was used to characterize the fracture energy. The transition temperature was taken as that at which the fracture energy was 2.7 J (2 ft-lb).

Fracture surfaces were examined by scanning electron microscopy (SEM). Optical metallography was facilitated by use of a saturated solution of picric acid in 100 ml water with 1 g sodium tridecylbenzene sulfonate added as a wetting agent.\(^11\) Immersion etching was done at room temperature for 2 min for all except as-quenched specimens, which were etched for 45 min.

Auger electron spectroscopy (AES) was used on specimens fractured at \(-90^\circ\)C in ultra high vacuum to determine intergranular compositions. The following conditions prevailed:

- **system:** PHI single pass CMA
- **ambient pressure:** ~10\(^{-8}\) Pa (6 \(\times\) 10\(^{-11}\) Torr)
- **beam current:** 50 \(\mu\)A
- **beam energy:** 2000 eV
- **spot size:** 500 \(\mu\)m
- **sweep rate:** 4 eV/s
- **lock-in ampl. sens.:** 100 \(\mu\)V
- **time constant:** 10 ms

Spectra were recorded from at least 5, and usually 10, areas on the fracture surface. Subsequent observation by SEM was used to determine the percent intergranular fracture on the AES specimens. The CHT specimens exhibited 100 pct intergranular fracture (Cf. Fig. 5); the IHT specimens appeared to give almost 100 pct intergranular fracture (Cf. Figs. 8 and 9). The values of at. pct P were obtained with the calibration data of Suzuki,\(^16\) who determined that for a homogeneous alloy the P concentration is 0.18 times the peak height (in pct of the 703 eV Fe peak) for the spectrometer conditions used in the present study.

**RESULTS**

The embrittlement behavior of the CHT steel is compared with that of the IHT steel in Fig. 2. The CHT steel exhibits: 1) a marked increase in the degree of embrittlement with increase in prior austenite grain size, 2) a steady state level of embrittlement achieved within 500 h at 480\(^\circ\)C, corresponding with the attainment of a constant intergranular concentration of P within 50 h, and 3) 'overaging' peaks (maxima in the embrittlement vs time curve) corresponding with the small decrease in hardness during aging. Except for the grain size effect, these features were observed previously in the same steel by Mulford et al.\(^9\) In contrast, the IHT steel shows: 1) only a slight transition temperature shift due to aging, 2) only a very small dependence on prior austenite grain size (i.e., prior to the intercritical heat treatment), and 3) substantially less P concentration on the fracture surfaces. There is essentially no difference in hardness between the CHT and IHT steels after 500 h aging, so the difference in behavior is not connected with the hardness factor.

The mean values and standard deviations of the P Auger peak height ratios on the intergranular fracture surfaces are given in Table II. The standard deviation generally increases with prior austenite grain size. This is presumably a reflection of a real variation in P concentration from one grain boundary to another, a phenomenon which is currently under study.\(^17\)

The effect of prior austenite grain size is shown more clearly in Fig. 3. In both the CHT and IHT steels, the transition temperature after 1000 h aging falls linearly with ASTM number, as noted earlier by Capus.\(^3\) However, in the CHT steel the slope is ~25\(^\circ\)C per unit,\(^*\) while in the IHT it is only ~5\(^\circ\)C per unit. As shown from the intergranular composition data in Fig. 2, the grain size effect is not attributable to a dependence of intergranular P concentration on grain size.

The special picric acid etchant proved invaluable in the microstructural characterization of this steel.

*This compares with ~10\(^\circ\)C per unit found by Capus,\(^3\) who used a different test method, hardness, aging time, and bulk P content, all of which would be expected to influence the slope.