**Table II. Comparison of Theoretical and Experimental $K$ Values in Aluminum, Nickel, and Copper**

| Metal | $T$ (°C) | $T/T_M$ | $K_E$ ($m^2/s$) | $K_T$ ($m^2/s$) | $K_E/K_T$ | SFE ($mJ/m^2$) |
|---|---|---|---|---|---|---|
| Aluminum | 250 | 0.56 | $5.92 \times 10^{-16}$ | $5.44 \times 10^{-16}$ | 1.09 | 200 |
| Nickel | 350 | 0.36 | $6.38 \times 10^{-17}$ | $4.62 \times 10^{-26}$ | $1.38 \times 10^{8}$ | 128 |
| | 300 | 0.33 | $7.4 \times 10^{-18}$ | $3.66 \times 10^{-28}$ | $2.03 \times 10^{10}$ | — |
| | 250 | 0.30 | $3.0 \times 10^{-18}$ | $1.14 \times 10^{-30}$ | $2.63 \times 10^{12}$ | — |
| | 200 | 0.27 | $5.18 \times 10^{-18}$ | $1.04 \times 10^{-33}$ | $4.98 \times 10^{15}$ | — |
| Copper | 200 | 0.35 | $5.7 \times 10^{-18}$ | $1.5 \times 10^{-25}$ | $3.8 \times 10^{7}$ | 78 |

$T_M$ = melting point in K; $K_T$ = theoretical $K$ value; $K_E$ = experimental $K$ value.

**Table III. The Observed Activation Energy Values and the Relative Contributions of the Sandstrom and Pipe Diffusion Models**

| | | Relative Contributions | |
|---|---|---|---|
| | | Sandstrom | Pipe Diffusion |
| $T$ (°C) | $Q$ (kJ/mole) | $Q$ = 292 kJ/mole | $Q$ = 126 kJ/mole |
| 200 | 150 | 0.16 | 0.84 |
| 250 | 168 | 0.25 | 0.75 |
| 300 | 179 | 0.32 | 0.68 |
| 350 | 183 | 0.34 | 0.66 |

homologous temperatures, as indicated in Table II. This may be related to the process of extraction and/or emission of dislocations from the subgrain boundaries.

Thus, we can summarize our results of this study in the following manner:

1. The parabolic equation, established by Sandstrom, to describe the subgrain growth is valid in nickel during recovery at 350 °C, 300 °C, 250 °C, and 200 °C.
2. There are large differences between the theoretical and the experimental $K$ values at these four recovery temperatures. The differences can be rationalized on the basis of two different mechanisms: subgrain growth due to (a) the vacancy mechanism, expected to dominate the process at higher temperatures, and (b) the dislocation pipe diffusion mechanism, expected to dominate the process at lower temperatures.

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# Effect of Prior Austenitic Grain Size on Stress Corrosion Cracking of a High-Strength Steel


G.F. LI, R.G. WU, and T.C. LEI


High-strength steels are greatly susceptible to stress corrosion cracking (SCC) and hydrogen embrittlement (HE). It was found that in 4340, 4330M, *etc.* steels, threshold stress intensity, $K_{Iscc}$ (or $K_{th}$), increased, apparently when specimens were quenched at higher temperatures.[1-4] However, different studies obtained different conclusions on the mechanism. The increase of $K_{Iscc}$ (or $K_{th}$) was attributed to the increase of the amount of retained austenite,[1] the growth of grains,[2,3] or the reduction of impurity segregation at grain boundaries.[4]

In this study, the variations of segregation at grain boundaries, grain size, amount of retained austenite, and other metallurgical factors of $30Cr_3SiNiMoV$ high-strength steel with quenching temperature were examined in order to determine which factor played a predominant role in the increase of $K_{Iscc}$. The chemical composition of the steel produced by induction melting and electric slag remelting is listed in Table I.

The specimens were austenitized in salt baths of 870 °C, 900 °C, 970 °C, 1100 °C, 1150 °C, and 1200 °C for 20 minutes and oil quenched. Then, all specimens were tempered at 300 °C for 2 hours.

Stress corrosion cracking tests were carried out by using modified wedge-opening load (WOL) specimens with


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Manuscript submitted May 17, 1989.

**Table I. Chemical Composition of the Steel (Weight Percent)**

| C | Si | Mn | P | S | Ni | Cr | Mo | V |
|---|---|---|---|---|---|---|---|---|
| 0.32 | 0.86 | 0.69 | 0.014 | 0.001 | 1.09 | 2.86 | 0.34 | 0.08 |

thicknesses of $B$ = 13 mm, which, loaded with bolts, were immersed in 3.5 pct NaCl aqueous solution at ambient temperature. When SCC stopped propagation, the threshold stress intensity, $K_{Iscc}$, was calculated in terms of loading displacement and final length of crack. The yield strength, $\sigma_{ys}$, was measured by using smooth tensile specimens.

The microstructure of the specimens was examined with transmission electron microscopy (TEM). The amount of retained austenite was measured with X-ray diffraction. The fracture surfaces of SCC were examined with scanning electron microscopy (SEM). Because the intergranular fracture surfaces of SCC showed the prior austenitic grains clearly, the average diameters of grains, $d$, were measured statistically on the fracture surfaces.

The segregation at prior austenitic grain boundaries was examined by using a PHI595 scanning Auger microprobe (SAM). In order to obtain intergranular fracture, Auger specimens were precharged cathodically with hydrogen and then fractured slowly in the ultrahigh vacuum chamber of the SAM when vacuum was better than $2.7 \times 10^{-7}$ Pa. Atomic ratios at grain boundaries were estimated in terms of the Auger peak heights and the sensitivity factors of elements.

All fracture surfaces of SCC in this steel were primarily intergranular along the prior austenitic grain boundaries, as shown in Figure 1. Figure 2 shows the variations of $K_{Iscc}$, $\sigma_{ys}$, prior austenitic grain diameter, $d$, retained austenitic amount, $A_r$, and the concentrations of carbon and phosphorus at grain boundaries with quenching temperature. With the rise of quenching temperature, threshold stress intensity, $K_{Iscc}$, increased steadily as did the diameter of prior austenitic grain, while the variation of yield strength was not apparent.

All Auger specimens were fractured primarily along prior austenitic grain boundaries. The analysis was conducted on the specimens quenched at 900 °C, 1100 °C, and 1150 °C. Figure 3 shows a typical Auger spectrum from the specimen quenched at 1150 °C. The main elements at the grain boundaries were Fe, C, Cr, and P, in which C and P were the elements segregated seriously as compared with the bulk chemical composition of the steel. The peak shape of carbon shows that the carbon at the grain boundaries exists primarily in the form of carbide.[5] Similar results were obtained from the specimens quenched at 900 °C and 1100 °C. The concentrations of C and P (the average of that at several intergranular surfaces) are shown in Figure 2 which shows little difference among the specimens quenched at different temperatures.

Analysis with TEM showed that the microstructures were primarily lath martensite with $\varepsilon$-carbide, with thin films of retained austenite between the martensitic laths. There were a few undissolved carbide particles in the specimens quenched at 870 °C and 900 °C. A few twins were also found in some zones when the quenching temperature was below 1100 °C. X-ray diffraction showed that the variation of retained austenitic amount was not

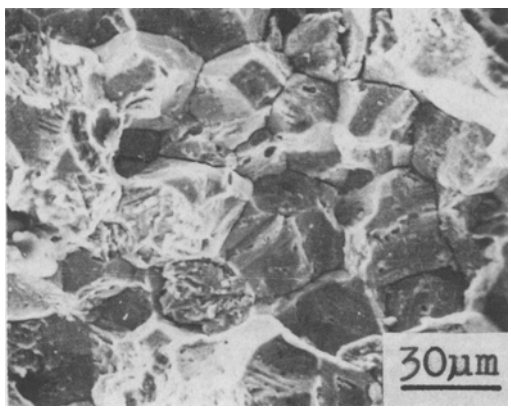


Fig. 1—Fracture surface of the specimen quenched at 970 °C.

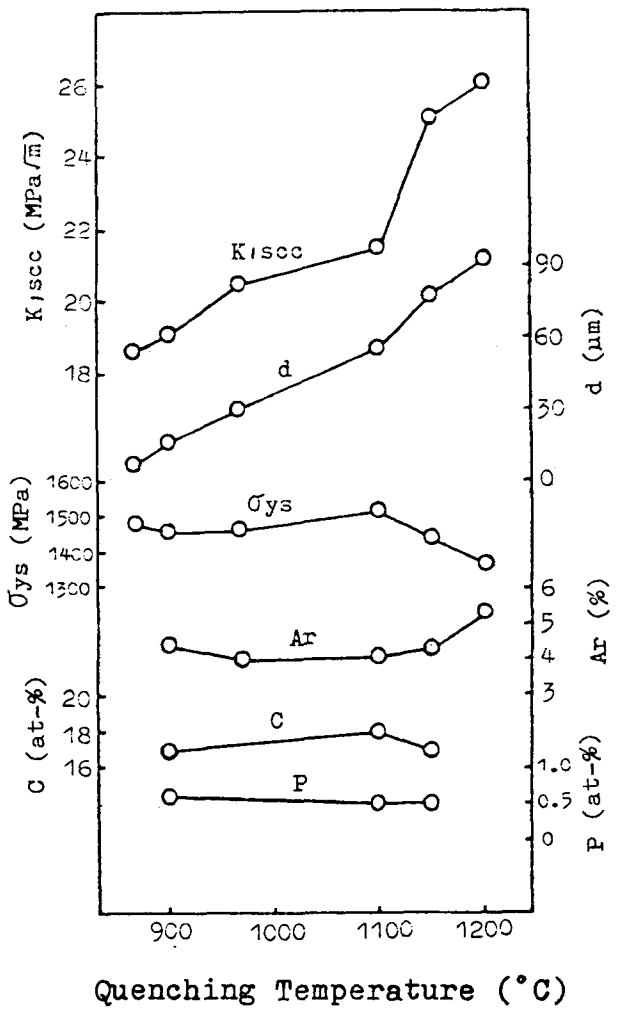


Fig. 2—Variations of $K_{Iscc}$, $\sigma_{ys}$, and some metallurgical factors with quenching temperature.

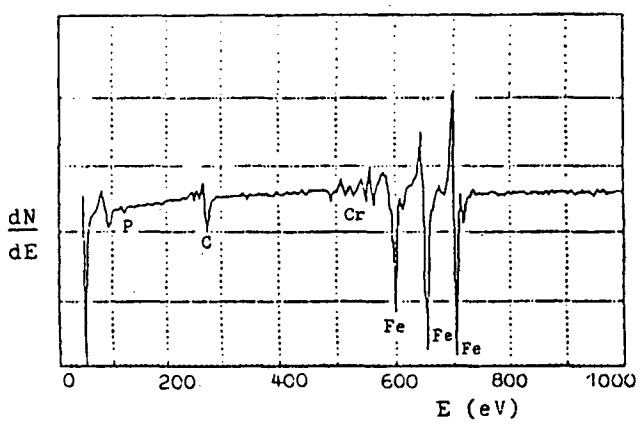


Fig. 3—A typical Auger spectrum from the intergranular surface of the specimen quenched at 1150 °C.

apparent when the quenching temperature varied in the range of 900 °C to 1200 °C, as shown in Figure 2.

Experimental results showed carbide and phosphorus which were segregated at the grain boundaries. This can be considered as main metallurgical factors causing intergranular fracture of the high-strength steel in a stress corrosion condition, because the interface between the carbide and the matrix can strongly trap hydrogen[6] produced in the stress corrosion process. Also, the phosphorus causes the reduction of cohesion of grain boundaries. However, the increase of $K_{Iscc}$ in this study cannot be attributed to the change of the segregation at grain boundaries, because no apparent difference was found in the segregation among specimens quenched at different temperatures. The variations of yield strength and retained austenite were also not apparent. The undissolved carbide and twins were few and existed primarily in the specimens quenched at lower temperatures. Therefore, it is hard to explain the steady increase of $K_{Iscc}$ with the rise of quenching temperature by means of their actions.

Only prior austenitic grain size was the factor which varied in step with $K_{Iscc}$. The steady growth of grains with the rise of quenching temperature should be the main reason causing the increase of $K_{Iscc}$. Figure 4 shows the

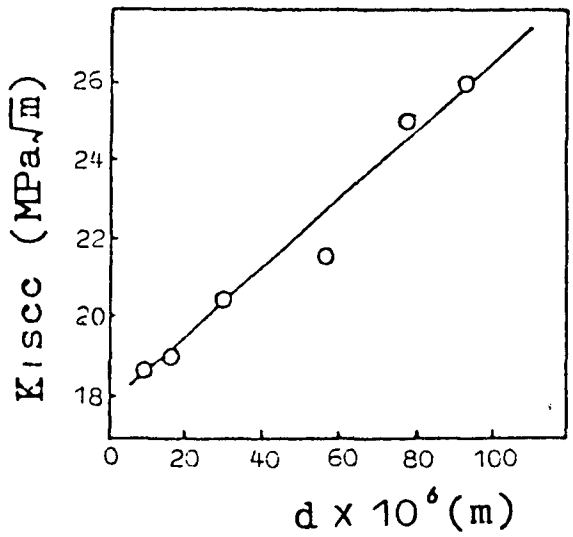


Fig. 4—Variation of $K_{Iscc}$ with grain diameter, $d$, of the steel.

variation of $K_{Iscc}$ with grain diameter, $d$. The equation of the straight line is

$$K_{Iscc} = 17.7 + 8.85 \times 10^4 d$$

Therefore, for intergranular fracture, the increase of grain size can be regarded as a factor raising the resistance of high-strength steels to stress corrosion cracking.

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