# Intergranular Corrosion Behavior of AISI 304 Stainless Steel

RAGHUVIR SINGH, B. RAVIKUMAR, A. KUMAR, P.K. DEY, and I. CHATTORAJ

The effects of prior cold rolling of up to an 80 pct reduction in thickness on the sensitization-desensitization behavior of Type AISI 304 stainless steel and its susceptibility to intergranular corrosion have been studied by electrochemical potentiokinetic reactivation (EPR) and Strauss-test methods. The results indicate that the prior deformation accelerated the sensitization as compared to the undeformed stainless steel. The deformed Type 304 stainless steel experienced desensitization at higher temperatures and times, and it was found to be enhanced by increased cold deformation. This could be attributed to the increased long-range chromium diffusion, possibly brought on by increasing pipe diffusion and vacancies. The role of the deformation-induced martensite (DIM) and texture, introduced by uniaxial cold rolling, on the sensitization-desensitization kinetics has also been discussed. This study could not reveal any systematic relationship between texture and the degree of sensitization (DOS) obtained. The effect of DIM on DOS seems to be pronounced at 500 °C when the steel retained significant amounts of DIM; however, the retained DIM is insignificant at higher sensitization times and temperatures.

## I. INTRODUCTION

STAINLESS steels are a common material of construction, as they offer a wide range of corrosion resistance, apart from good fabrication and mechanical properties, to many industrial environments (*e.g.*, chemical, fertilizer, and nuclear industries). At the same time, these alloys are prone to microstructural changes, along with variations in chemistry, when exposed to sensitization temperatures due to faulty heat treatment or welding operations (especially in the heat-affected zones). In the case of AISI 304/316 stainless steels, precipitation of chromium carbides takes place along the grain-boundary region in the temperature range of 480 °C to 815 °C. This results in chromium depletion near the grain boundary, and the resultant grain-boundary region is prone to preferential intergranular corrosion (IGC)[1,2,3] in several environments. These sensitized steels, when subjected to load, experience crack propagation through the boundary, leading to even faster failures, and are, therefore, responsible for the premature collapse of several engineering components.

Prior to shaping the component into the desired geometry, steels often undergo a thermomechanical processing, during which materials change certain properties. By interaction of the temperatures and strains encountered during thermomechanical processing, a material's microchemistry may be altered so that its functional properties are changed. Cold deformations are known to alter the properties of steels by increasing the dislocation density, which, apart from its effect on mechanical properties, may help in accelerating diffusion of certain species, phase transformation, nucleation, *etc*. There have been contradictions in the literature about the net influence of deformation on the corrosion of sensitized stainless steels.[2–5] Briant and Ritter observed transgranular attack in Type 304 stainless steel, which they assumed to be due to deformation-induced martensite (DIM); however, similar corrosion attack has been noted in the deformed Type 316 stainless steel, that does not consist of such a dual phase.[6] The state of deformation (uniaxial tensile, compressive, or cross rolling) itself may be a variable that can influence the end results.[7] In spite of several studies performed on the prior-deformation effect on sensitization and IGC, a universally accepted conclusion is yet to be made. The reasons of such conflicting results could be due to the consideration of limited strains (either tensile or compressive),[4,6,8,9] temperatures,[8,10,11] or times[7,10,12] as variables in such studies. In this study, we use a wide range of the aforementioned variables.

The grain-boundary effect, which governs the sensitization behavior, is reported to depend on the nature of the boundary. Geometrically, the grain boundaries are classified by the misorientation axis and angle with respect to neighboring grains. The misorientation angle and atomic structures in the boundary region have been observed to play an important role in the sensitization kinetics.[13,14,15] Mechanical deformation, which imparts crystallographic texture in the materials, is a viable route to change these characteristics; the correlation of sensitization mechanism with the latter is expected. A few studies have appeared on the textural effects on corrosion kinetics;[16] however, there is none which address sensitization in light of the texture development. In the present work, investigations have been carried out on a wide range of plastic deformations and on their effects on sensitization and the IGC behavior of AISI 304 stainless steel. Texture development as a result of mechanical deformation was also evaluated for its possible role in sensitization.

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Manuscript submitted October 31, 2002.

## II. EXPERIMENTAL TECHNIQUES

The experiments were conducted on AISI 304 stainless steel containing 0.05 pct C, 18.54 pct Cr, 9.8 pct Ni, 1.8 pct Mn, and 0.54 pct Si by weight. Plates of AISI 304 stainless steel, 0.8-cm thick, were solution annealed at 1100 °C for 1 hour and cut into various plate sizes for the cold-rolling operation. The stainless steel plates were processed by unidirectional cold rolling, impressing 20, 40, 60, and 80 pct reductions in thickness, at room temperature. Samples with dimensions of 2.0 × 3.0 cm × thickness were obtained from each cold-rolled plate. These samples were then subjected to aging at 500 °C, 600 °C, and 700 °C for different time periods. Transformation-induced martensite, formed during cold rolling, was determined by X-ray diffraction. The texture measurement on the rolled surface was made by an X-ray diffractometer using the Schultz reflection technique.[17] For texture measurement and martensite determination, specimens measuring 2.5 × 3.0 cm were cut and electropolished in a solution of 125 mL $H_2SO_4$ + 650 mL orthophosphoric acid in 225 mL distilled water. Polishing was performed at 85 °C ± 2 °C under controlled current and potential conditions, with a copper rod used as a cathode.

The degree of sensitization was determined by the double-loop electrochemical potentiokinetic reactivation (EPR) tests. Samples were polished using up to 1200-grit emery papers, followed by degreasing in acetone solutions. The test solution, *i.e.*, 0.5M $H_2SO_4$ + 0.01M KSCN, was prepared from reagent-grade chemicals in distilled water. Before polarizing, the samples were cathodically cleaned at a potential of −900 mV with respect to a saturated calomel electrode (SCE) for 120 seconds. Scanning was initiated at a potential of −100 mV (with respect to the open-circuit potential (OCP)) and reversed at a potential of +300 mV (SCE) at a scan rate of 6 V/h. The SCE was used as a reference electrode. The degree of sensitization (DOS) was evaluated by measuring the ratio of $I_r/I_a$, where $I_r$ was the reactivation current density and $I_a$ was the activation current density. Modified Strauss tests were conducted on the samples as per ASTM specifications,[18] with a reduced test period of 36 hours. The corrosion rates were obtained by using the formula given as follows[12,18] and are reported in millimeters/month.

$$\text{Corrosion rate (mm/month)} = \frac{7290\,W}{A \times d \times t}$$

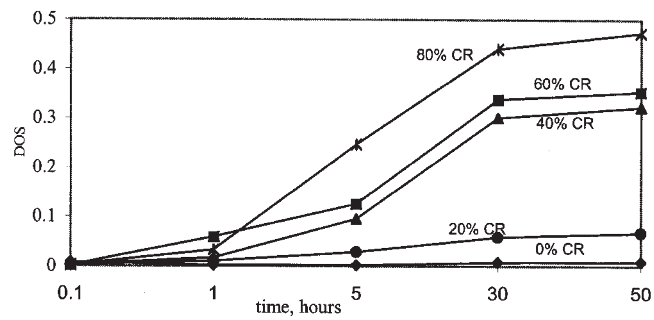
Where:

$W$ = weight loss (grams),
$A$ = area of specimen ($cm^2$),
$d$ = density ($g/cm^3$) (for AISI 304 stainless steel, $d = 7.9\ g/cm^3$), and
$t$ = time of exposure (hours).

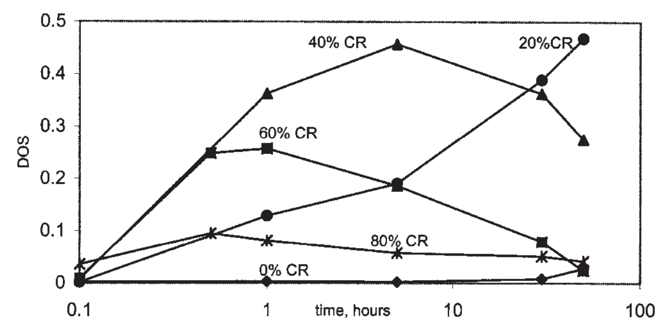
## III. RESULTS

### A. *Degree of Sensitization*

In undeformed samples, the DOS was found to increase with aging temperature and time. A DOS value higher than 0.05 generally corresponds to what is classically termed a "ditch structure," which alludes to the grain-boundary appearance after an oxalic acid etch test.[18] Under such conditions, the material is considered unsuitable for applications. For the undeformed samples, such deteriorations were observed only in a few sensitization conditions (time/temperature combinations) like 700 °C/30 h and 700 °C/50 h. The effect of cold working on the sensitization behavior is demonstrated in Figures 1(a) through (c). First, it has to be appreciated that for the temperatures and times of sensitization studied, prior cold working did enhance sensitization susceptibility. The DOS at 500 °C (Figure 1(a)) appears to increase with cold deformation. For higher temperatures of sensitization, as shown in Figures 1(b) and (c), the DOS is not a monotonically increasing function of the amount of cold deformation. For instance, for samples sensitized at 600 °C (Figure 1(b)), those that were prior cold rolled by 40 pct showed higher sensitization at all times of sensitization as compared to those cold rolled by 60 or 80 pct. In fact, a maximum in DOS with regards to the percentage of prior cold reduction is obtained for any given sensitization condition. The other important aspect, which is evident from




(*a*)

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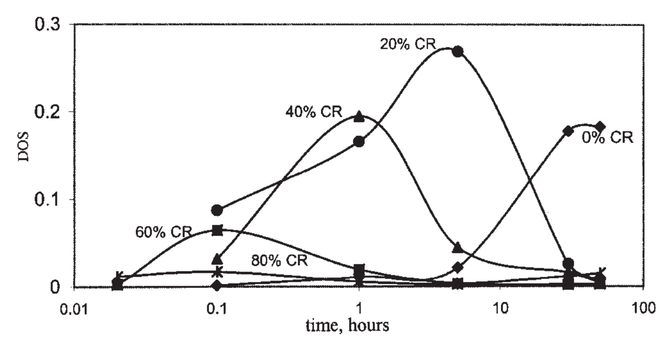


(*c*)

Fig. 1—(*a*) through (*c*) Effect of deformation on DOS values at (a) 500 °C, (b) 600 °C, and (c) 700 °C with varying aging time.

Figures 1(b) and (c), is that prior cold deformation promotes self-healing or desensitization, a phenomenon in sensitized steels whereby it regains its corrosion resistance. This is brought about by the competition between chromium and carbon diffusion[19,20,21] and is discussed in detail in 'Discussion' section. At the lowest sensitization temperature of 500 °C, desensitization was not observed. For the other sensitized samples, the self-healing time decreased with higher temperatures of sensitization. This temperature effect is a direct result of the dependence of volume diffusion (of chromium) on the absolute temperature and has been previously reported. Our results show that prior cold deformation can also cause an enhancement of the self-healing above and beyond the temperature effect. The self-healing time required for the different cold-worked samples at different temperatures was measured from the time of sensitization corresponding to the maximum DOS. These are shown in Figure 2. The inverse of desensitization time ($1/t_s$) is plotted in the abscissa in this figure. No self-healing occurred at 500 °C for up to 50 hours of annealing for any of the samples. An inverse relationship between the amount of prior cold work and the desensitization time is observed for both the sensitization temperatures, *i.e.*, 600 °C and 700 °C. Thus, prior cold deformation does enhance the long-range diffusive and mass-transfer processes (of chromium) responsible for the replenishment of Cr near grain-boundary-depleted zones. This is discussed in greater detail in 'Discussion' section.

The microstructural evidence obtained from samples exposed to the EPR test substantiates the role of prior deformation on desensitization, as shown in Figures 3(a) through (d), which shows the appearance of four EPR-tested samples which were subjected to different amounts of prior cold rolling and then

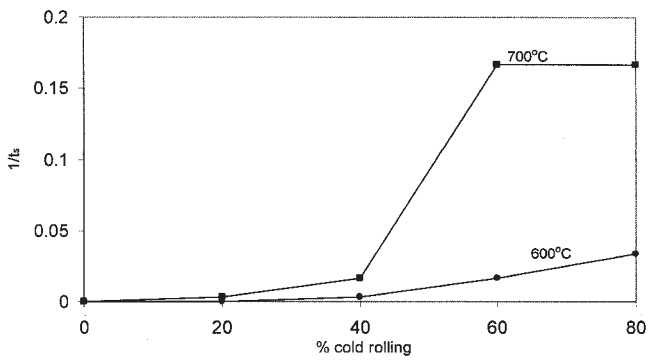


Fig. 2—Variation in desensitization time at different temperatures with degree of cold rolling.

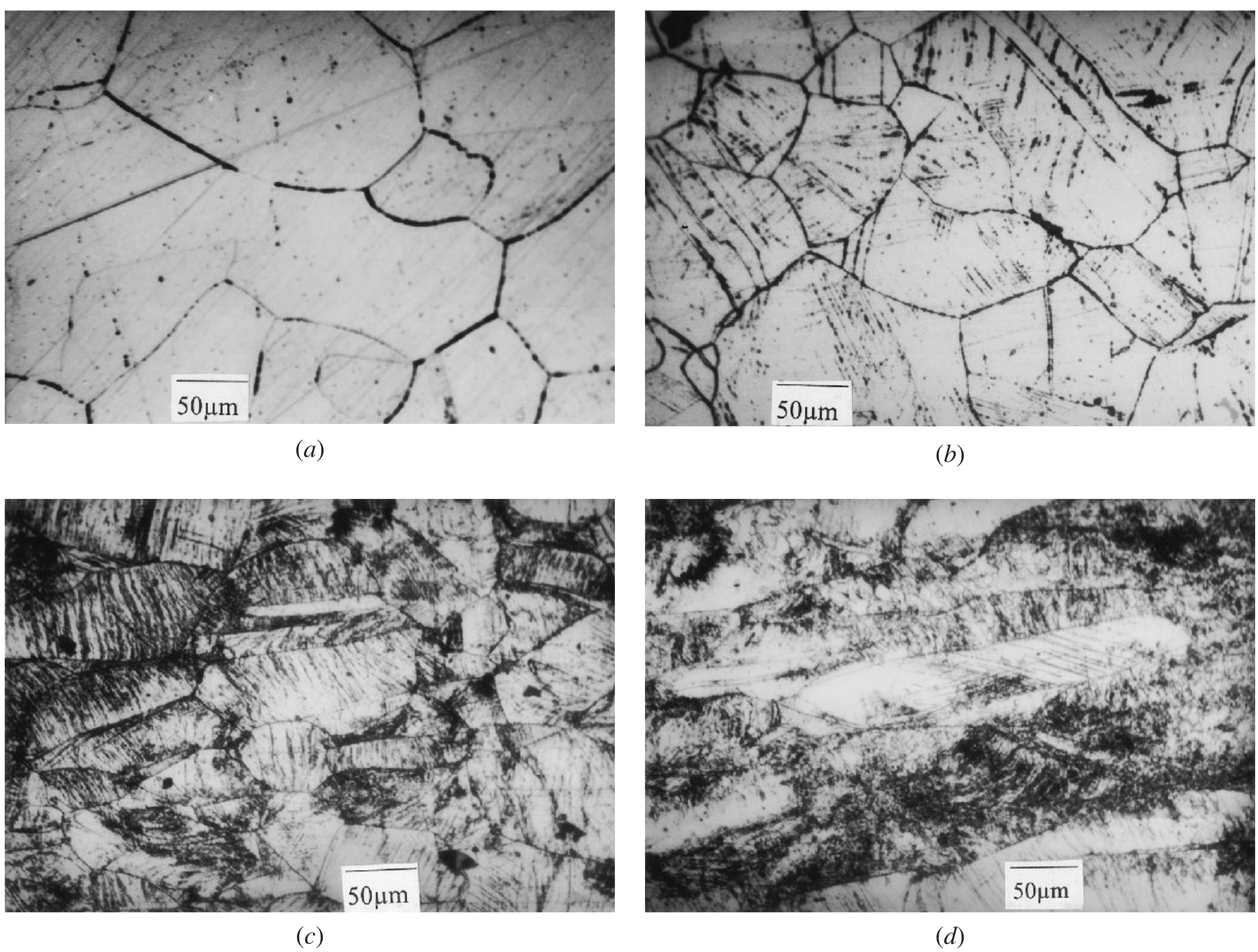


Fig. 3—(*a*) through (*d*) Microstructures of the post-EPR specimens, sensitized at 700 °C for 5 h after (a) homogenization and after cold rolling to (b) 20 pct, (c) 40 pct, and (d) 60 pct, reductions in thickness.

exposed to the same sensitization condition. It was observed that the extent of grain-boundary attack was at a maximum for samples with the intermediate prior cold rolling (Figure 3(b)) and that in the extensively cold-rolled sample, the corrosive attack was diffuse with no preferential attack of the grain boundaries. This is similar to the observation of transgranular corrosion at higher deformation, made by other authors.[4,6,12]

B. *Modified Strauss test*

The results of the modified Strauss tests corroborate the findings of the previous sections with regards to DOS. The propensity of the IGC is represented in terms of corrosion rate, which is presumably a result of localized attack confined along the grain boundary. The corrosion rates (in millimeters/month), obtained from these experiments for some of the sensitized coupons are reported in Table I. Like the EPR test results, the undeformed stainless steel follows an increase of the corrosion rate with experimental time/temperature; for the deformed specimens, the exhibition of maxima is in keeping with the observations on DOS.

C. *Texture correlation with DOS*

The role of texture was investigated for its possible effect on sensitization. The (111) pole figures obtained from homogenized and various rolled specimens are presented, using standard projection, in Figures 4(a) through (c). The pole figures illustrate the texture with the main components, whose intensity is found to increase, gradually, with degree of cold rolling. The pole-figure results showed that at 20 pct cold rolling, {110}<112> and {011}<100> were the major texture components. The textural evolution after 40 pct cold rolling is similar to that of 20 pct cold rolling, except that the sharpness increases. With an increase in cold deformations (60 and 80 pct), the {110}<112> component becomes prominent and a weak {111}<110> component appears. The texture developed after 80 pct cold rolling is similar to that observed in brass. The texture evolution with cold working does not show any correspondence with the variation of DOS. In our investigations, we are unable to comment on the possible role of texture on sensitization; a detailed investigation on the role of texture needs to be conducted, which was beyond the scope of the present work.

D. *Deformation-induced martensite and DOS*

The cold rolling is found to induce a martensitic phase in Type 304 stainless steel. This was found to increase with severity of deformation, as determined by X-ray diffraction and shown in Table II. It was observed that at a deformation of 80 pct, the martensite is a dominant phase in Type 304 stainless steel. The evolution of the martensitic phase in the material is also collaborated by an increase in hardness of the cold-worked steels. The presence of martensite is potentially significant with respect to the sensitization process in two ways. The carbon and chromium activities may be significantly different in this phase as compared to austenite. The chromium diffusivity would also be significantly different in a martensitic phase as compared to an austenitic phase. However, the DIM is a metastable phase, which decreases with temperature.[22,23] It was reported that half of the DIM reversion takes place at 550 °C ± 20 °C, and it is almost completely reversed within few minutes of exposure at 750 °C. At any time, the maximum amount of the martensite in the deformed Type 304 stainless steel will persist at 500 °C and reduce to almost negligible amounts when sensitized at 700 °C. Thus, for our samples, the effect of DIM on sensitization would most likely be restricted to the low temperature/time combinations. This was verified by measurements on sensitized samples, some of which are listed in Table II. The martensite was transformed to austenite, and no evidence of ferrite was found. It was observed that after sensitization treatments done at 500 °C, significant amounts of DIM were retained in the cold-worked samples, especially those which were extensively cold worked. The monotonic increase in DOS with cold work, for this sensitization temperature, can be attributed to the higher amounts of DIM. Such correlations have been reported.[4] However at higher sensitization temperatures,

**Table I. Corrosion Rates (mm/Month) from Strauss Tests**

| Deformation (pct cold Rolling) | 700 °C/ 0.2 h | 700 °C/ 1 h | 700 °C/ 5 h | 600 °C/ 1 h | 600 °C/ 5 h | 500 °C/ 5 h |
|---|---|---|---|---|---|---|
| 0 | 0.6 | 2.7 | 6.9 | 0.2 | 5.9 | 0.2 |
| 20 | 1.3 | 5.4 | 6.7 | 2.4 | 5.2 | 0.3 |
| 40 | 1.2 | 4.1 | 1 | 3.6 | 3.2 | 0.5 |
| 60 | 0.4 | 0.5 | 0.4 | 1.9 | 2.4 | 0.4 |
| 80 | 0.2 | 0.4 | 0.2 | 0.8 | 0.4 | 0.3 |

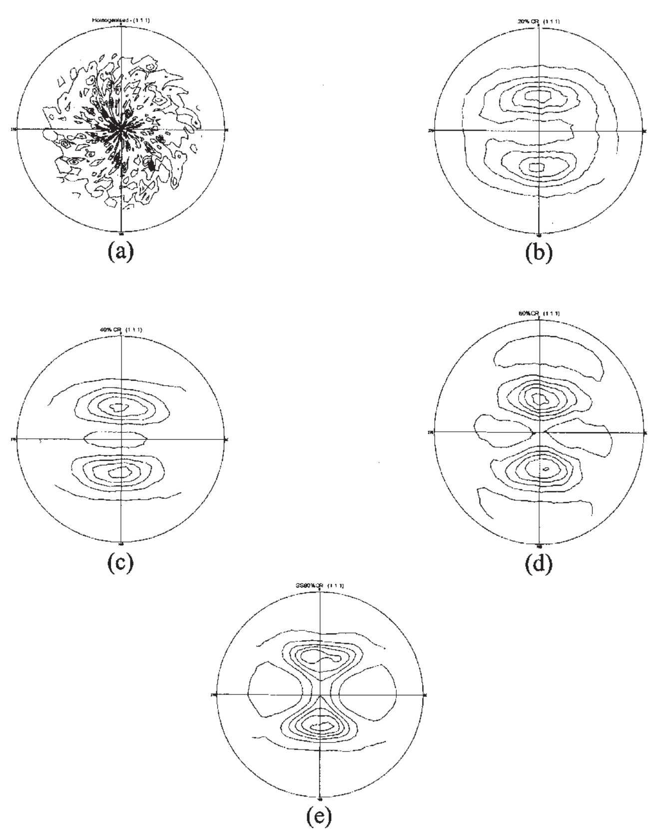

Fig. 4—(*a*) through (*e*) Pole figures of SS 304 (*a*) after homogenization, and after cold rolling to (*b*) 20 pct, (*c*) 40 pct, (*d*) 60 pct, and (*e*) 80 pct reductions in thickness.

the samples with the highest amounts of DIM retained after sensitization are not necessarily the ones with the higher DOS. The process of desensitization, which is accelerated both by higher temperatures as well as by higher amounts of deformation, counteracts the detrimental influence of DIM percentage on sensitization.

## IV. DISCUSSION

The observations made during this investigation have clearly indicated a definite effect of prior cold work on IGC sensitivity through its effect on the sensitization process. As we have indicated, a definite correlation of texture with sensitization susceptibility could not be found in these studies. There is strong indication that the primary influence of cold deformation is through its effect on the chromium mass-transfer process. An elaboration of the sensitization and subsequent self-healing or desensitization is necessary. The process of sensitization involves grain-boundary chromium carbide formation leading to a near-grain-boundary chromium depletion. The carbon diffusion is significantly faster than chromium diffusion, so that while there is a near-grain-boundary depletion of chromium, the carbon reduction is global and, therefore, insignificant. The process of desensitization has been very well explained by Stawstrom and Hillert.[21] As a first approximation, they had considered that the sensitization process was completed prior to desensitization, and, using chromium mass balance, they had come up with the following expression for the time for desensitization ($t_s$):

$$t_s = \frac{1}{D_{\mathrm{Cr}}^{\mathrm{o}}} \left( \frac{a X_{\mathrm{c}}^{\mathrm{o}}}{X_{\mathrm{Cr}}^{\mathrm{o}} - 0.13} \right)^2 \qquad [1]$$

where $a$ is the grain size, $X_{\mathrm{c}}^{\mathrm{o}}$ is initial carbon content, $X_{\mathrm{Cr}}^{\mathrm{o}}$ is initial chromium content, and $D_{\mathrm{Cr}}^{\mathrm{o}}$ is the diffusion coefficient of chromium. Their more rigorous treatment of desensitization considered simultaneous occurrence of the precipitation process as well as long-range chromium replenishment (self-healing). However, for low carbon activities, as is generally found in stainless steels, Eq.[1] was found to be very close to the results of the rigorous treatment. It may be seen from these findings that the two variables which define the self-healing are the grain size and the chromium diffusivity ($a^2/D_{\mathrm{Cr}}^{\mathrm{o}}$). We will discuss how cold deformation affects either of these variables.

The change in the grain size is a natural consequence of cold rolling, and these changes are tabulated in Table III. The cold-worked grains are naturally nonequiaxed, and the size reported is the length of the shortest distance between grain boundaries, as we are concerned with diffusive lengths. It is evident from Table III that there was a progressive decrease in the grain size in the thickness direction (YZ and ZX planes) with increase in cold rolling. There was no evidence of recrystallization on sensitization, and the grain sizes were not significantly affected by the sensitization treatment. The difference in grain sizes were less than 2 times between the two extremes of rolling (0 and 80 pct), which clearly would contribute only insignificantly to the decrease in the time of sensitization. The latter showed orders-of-magnitude change on cold rolling, as depicted in Figure 2, which cannot be accounted for by the change in grain size.

The chromium diffusivity may be affected by cold deformation in various ways. The transformation of austenite to martensite due to deformation would affect Cr diffusivity due to different crystallographic nature of the two phases.

**Table II. Deformation Induced Martensite Retained after Sensitization**

| | | Pct Deformation Induced Martensite* | | | | | |
|---|---|---|---|---|---|---|---|
| | | Sensitization Condition (Temperature/Time) | | | | | |
| Pct Cold Rolling | Hardness, $H_v$ | As Received | 500 °C, 30 h | 600 °C, 5 h | 600 °C, 30 h | 700 °C, 1 h | 700 °C, 30 h |
| 0 | 205 | 0 | 0 | 0 | 0 | 0 | 0 |
| 20 | 360 | 13 | 1.25 | 0.8 | 1.5 | 3.1 | 1 |
| 40 | 393 | 27 | 3 | 2 | 2.25 | 4.25 | 2 |
| 60 | 410 | 36 | 15.6 | 9 | 6 | 3.5 | 1.75 |
| 80 | 466 | 55 | 28.5 | 14.75 | 8.5 | 4 | 3.15 |

*Percent deformation induced martensite is determined within ±3 pct accuracy level.

**Table III. The Grain Sizes after Deformation and Sensitization; X is the Rolling Direction, Y is the Transverse Direction, and Z is the Thickness Direction**

| | Average Grain Size (mm) | | | | | |
|---|---|---|---|---|---|---|
| | Nonsensitized | | | Sensitized at 700 °C/50 h | | |
| Percent Rolling | XY plane | YZ plane | ZX plane | XY plane | YZ plane | ZX plane |
| 0 | 0.105 | 0.11 | 0.112 | 0.12 | 0.1 | 0.106 |
| 20 | 0.093 | 0.085 | 0.099 | 0.102 | 0.094 | 0.08 |
| 40 | 0.098 | 0.076 | 0.072 | 0.088 | 0.074 | 0.073 |
| 60 | 0.101 | 0.07 | 0.068 | 0.096 | 0.709 | 0.073 |
| 80 | 0.111 | — | — | 0.101 | — | — |

However, this effect is important only at low temperatures of sensitization, as discussed earlier. The more important aspect of deformation is the generation of vacancies, dislocations, and dislocation networks. It may be noted that the dislocations introduce the possibility of pipe diffusion, an alternate diffusion mechanism that complements the normal volume-diffusion process, so that the overall diffusivity of Cr may be represented by[24]

$$D_{\mathrm{Cr}} = D_{\mathrm{Cr}(i)} + nA_pD_p \quad [2]$$

where $D_{\mathrm{Cr}}$ is the bulk chromium diffusivity in the lattice, $D_{\mathrm{Cr}(i)}$ is the chromium diffusivity in the undeformed lattice, $n$ is the dislocation density, and $D_p$ is the diffusivity through a dislocation pipe of cross-sectional area $A_p$. If we assume that the grain-size contribution on $t_s$ is minimum, by combining Eqs.[1] and [2] we obtain the following relationship:

$$1/t_s \propto (D_{\mathrm{Cr}(i)} + nA_pD_p) = D_{\mathrm{Cr}(i)}(1 + nA_pD_p/D_{\mathrm{Cr}(i)}) \quad [3]$$

Further, if we assume that $A_p$ and $D_p$ remain reasonably constant with deformation, the time of sensitization is expressible in the following way:

$$1/t_s = K_1(1 + K_2n) \quad [4]$$

where $K_1$ and $K_2$ are temperature-dependent constants.

The dislocation density is an increasing function of the amount of plastic strain, and for steels, it is sigmoidal in form,[25] attaining saturation at high plastic strains. Since higher cold work implies higher plastic strain, the decrease in desensitization time is evident from Eq. [4]. This is also shown in Figure 2. The saturation in the $t_s$ values at high cold work is due to the saturation of the dislocation-density value. The temperature effect is due to the higher volume diffusion of chromium ($D_{\mathrm{Cr}(i)}$) at higher temperatures.

One of the most important mechanisms of diffusion for solutes is the so-called vacancy diffusion, and an enhancement of vacancy concentration directly influences the solute diffusivity. Vacancies are known to experience a manifold increase by cold work. However, subsequent high-temperature exposure (as in sensitization) brings about various mechanisms of vacancy annihilation that create voids or cavities at grain boundaries and slip bands, so that a concentration close to the equilibrium concentration for that temperature is attained. It has been shown by Mecking and Estrin[26] that supersaturation of vacancies generated by plastic deformation is negligible at temperatures close to and above 0.5 $T_m$. Thus, a deformation-induced vacancy concentration will have a significant effect on $D_{\mathrm{Cr}}$ values at low sensitization temperatures, so that the value of $1/t_s$ will monotonically increase with deformation, whereas at higher temperatures, this effect is negligible.

Thus, the effect of cold working on the desensitization process is largely due to its effect on bulk chromium diffusion through the introduction of dislocations and vacancies that facilitate mass transfer.

## V. CONCLUSIONS

The effect of prior cold deformation on subsequent sensitization and intergranular corrosion susceptibility was studied. Prior deformation was found to enhance sensitization as measured by the DOS and corroborated by Strauss tests, for all sensitization temperatures and times, as compared to the undeformed samples. However the DOS was not a monotonically increasing function of the amount of prior cold deformation at higher temperatures. This could be rationalized as being due to a competition between the short-range diffusive processes that result in sensitization and the long-range chromium diffusion that leads to desensitization. The process of desensitization was found to be accelerated by cold deformation. The cold deformation was found to introduce changes in texture, grain size, and the amount of DIM. There was no systematic correlation observed between the texture developed and the IGC susceptibility in our studies. The enhanced desensitization could be attributed partially, and to a small extent, to the reduced grain size (diffusive length) after cold rolling. The most important aspect of deformation vis-à-vis desensitization was the enhanced long-range chromium diffusion, which we feel was due to increased dislocation density and vacancies with increased cold rolling. Earlier studies on deformation effects on sensitization have neglected to address the issue of desensitization enhancement.

## ACKNOWLEDGMENT

The authors record their sincere thanks to Professor S.P. Mehrotra, Director, National Metallurgical Laboratory, for granting permission to publish this article. The authors are thankful to Dr. S. Ghosh Chowdhury for fruitful discussions on the texture. This work is funded by the Department of Science & Technology (New Delhi, India).

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