Study on the Effect of Slight Prestrain Annealing on Sensitization in the 316l Stainless Steel

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Abstract

This work deals to bring out the study on the effect of slight prestrain annealing during sensitization on an AISI 316L stainless steel and to simultaneously examine the Inter-Granular Corrosion (IGC) caused by sensitization. Initially, the 316L stainless steel is heated to 1000°C - 1200°C in a furnace for 60 minutes and is water quenched thereafter. Cold rolling is worked on the sample pieces. Finally, Intergranular Corrosion, microstructure and the texture of the 316L stainless steel is analyzed and the effect caused by different reduction percentage is studied by using a Scanning Electron Microscope (SEM analysis).

Keywords

Annealing, Sensitization, Deformation, Intergranular corrosion, Cold Rolling, SEM Analysis

I. Introduction

Austenitic Stainless Steels (ASS) have excellent resistance to general corrosion. They are, however, prone to localized corrosion – crevice and pitting corrosion, Intergranular Corrosion (IGC) and stress corrosion cracking (IGSCC). The two forms of localized corrosion namely, IGC and IGSCC, are often caused by sensitization - though the same can also be caused by segregation of active elements. Sensitization is typically created when an ASS is welded or heat treated for sufficient time in the temperature range of 1000-1200ºC, which leads to precipitation of chromium rich carbides at the grain boundaries. Growth of such carbides can lead to the formation of chromium depleted zones in the immediate surroundings. This makes the sensitized ASS prone to IGC and IGSCC.

The chief drawback in sensitization is the occurrence of Inter-Granular Corrosion (IGC). This type of corrosion arises typically in stainless steels. IGC can be reduced by using low carbon grade stainless steels such as 316L and 304L. In this experiment, AISI 316L stainless steel is used which contains 16% chromium, 10% nickel and 2% molybdenum. We are going to study the IGC of the material when it undergoes different reduction. This material comes under the standard industrial grade. The material was annealed (Water Quenched) and cold rolled to different reduction percentages (20-80%) by unidirectional rolling.

A. Deformation

All forming operations typically involve plastic deformation of metals. Understanding plastic deformation is important for perfecting such forming operations and also for modifying the structure, the latter being more relevant to the present study. In this section, different aspects of plastic deformation, based on their relevance to the present study, are looked at.

The main factors affecting plastic deformation in single-phase polycrystalline material can be generalized as:

- Structural parameters - slip-twin system(s), critical resolved shear stress (CRSS) and stacking fault energy (SFE) [25,26,55-58,100,101].
- Parameters related to the Crystals – Size and Shape [31,102] and Orientations [25-31].
- Deformation conditions – temperature, strain path and strain rate [19,28].

These factors can affect the deformation modes (relative activation of slip-twins) and developments of deformation texture and microstructure – the issues considered relevant to the present study.

B. Sensitization

It happens when a stainless steel is held at a temperature of 425 - 815°C and chromium carbides precipitate at the grain boundaries. This precipitation happens because the carbides are insoluble at these temperatures. In order for the carbide to precipitate, it must obtain chromium from the surrounding metal. This means that there is a chromium-depleted zone around the grain boundaries.. If a sensitized alloy is exposed to a corrosive environment, the areas near the grain boundaries are preferentially attacked. As the corrosion proceeds, the grains fall out and the metal loses strength.

C. Intergranular Corrosion

This situation can happen in otherwise corrosion-resistant alloys, when the grain boundaries are depleted, known as grain boundary depletion, of the corrosion-inhibiting elements such as chromium by some mechanism. In nickel alloys and austenitic stainless steels, where chromium is added for corrosion resistance, the mechanism involved is precipitation of chromium carbide at the grain boundaries, resulting in the formation of chromium-depleted zones adjacent to the grain boundaries (this process is called sensitization). Around 12% chromium is minimally required to ensure passivation, a mechanism by which an ultra thin invisible film, known as passive film, forms on the surface of stainless steels. This passive film protects the metal from corrosive environments. The self-healing property of the passive film makes the steel stainless.

II. Methodology

AISI 316 L austenitic stainless steel materials is chosen for the experiment. The material is cut with a dimension of 1.5x10 cm. The workpiece is then heat treated at a temperature of 1050-1200°C in a furnace for 60 minutes. It is removed from the furnace and immediately water quenched. The sample undergoes cold rolling process with various reduction of 20%, 60%, 80%. Finally, the Inter-Granular Corrosion is observed using a Scanning Electron Microscope.

A. Deformation

X-ray ODFs (Orientation Distribution Functions) were obtained by the standard series expansion method using the program MTM-FHM. Volume fractions, for individual ideal texture components, were estimated by convoluting the X-ray ODFs with suitable model functions, with an integrated ODF value of 1 and 16.5° Gaussian spread. The starting bulk textures (generalized as as-received texture) were discretized into 3000 representative orientations and were then subjected to Taylor type deformation texture simulations [18,19,25,27,28,42,55,118-124]. The results of texture measurements and simulations were analyzed and compared from the respective volume fractions of ideal texture components.

B. Corrosion

The degree of sensitization (DOS) was evaluated by the Double Loop Electrochemical Potentiokinetic Reactivation (DL-EPR) method. A fresh solution of 0.5 M H_2SO_4 + 0.01 M KSCN was used in the test. A platinum sheet and a saturated calomel electrode (SCE) were used as counter and reference electrodes respectively. The solution was de-aerated by passing argon gas before as well as during the tests and the tests were conducted at ambient temperature (26^0C). At least two samples were tested to ensure reproducibility of the results. The peak reactivation current (I_r) and the peak activation current (I_a) were measured during the backward and the forward scans respectively. The ratio (I_r/I_a)x100 was taken as a measure of the DOS. The material after (i) the annealing heat treatment and (ii) different percentages of cold deformation, annealing and sensitization heat treatment were tested by the DL-EPR test.

After the test, the sample was examined by a Scanning Electron Microscope (SEM).

III. RESULTS & DISCUSSION

A. Deformation

X-ray ODFs of the material after annealing were obtained by the standard series expansion method using the program MTM-FHM. Fig 3.1 shows the as-received undeformed material with maximum ODF intensity of 6.12. In fig 3.2, cold reduction substantially increased the texture-maximum ODF at 80%.

The result of ODFs, as in fig. 3.2, has been further elaborated in fig. 3.3, in terms of volume fractions of major texture components. The major texture components were identified from the usual ideal fcc texture components with, an average, more than 5% volume (approximate limit for effective X-ray detection). The changes in the texture components, with increasing percentage reduction, were assumed to be linear and the trends of such changes were estimated from the slope. As given in Table 3.1, Copper and Sulphur had more significant increase.

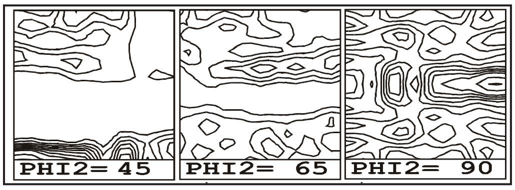

Fig 3.1: X-ray ODFs of type 316L stainless steel (Undeformed as-received)

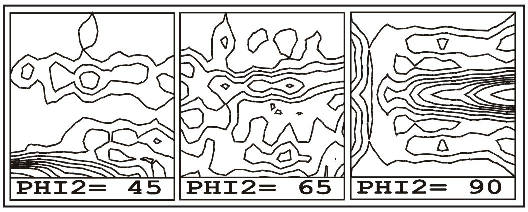

Fig 3.2. X-ray ODFs of type 316L stainless steel (80% reduction)

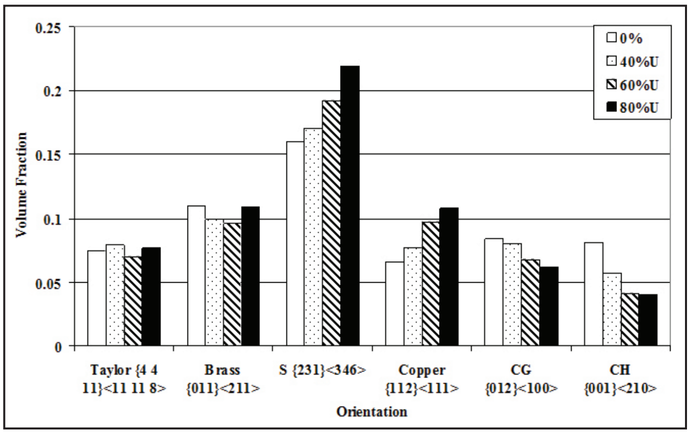

Fig 3.3: Estimated volume fractions (11° Gaussian spread) of the ideal texture components in type 316L stainless steel as a function of the deformation percentage

Table 3.1: Relative changes in major texture components, during deformation of type 316L stainless steel. The relative changes were identified from the respective slopes (of volume fractions against percentage reduction (as in fig. 3.3), which were assumed to be linear).

Major Orientations	Type 316L SS
	Rolling
Taylor {4 4 11}<11 11 8>	-0.001
Brass {011}<211>	-0.005
S {231}<346>	0.071
Copper {112}<111>	0.054
CG {012}<100>	-0.028
CH {001}<210>	-0.055

B. Corrosion

1. Degree of sensitization and intergranular corrosion

The DL-EPR ratio for the material after different degrees of cold deformation is tabulated in table 3.2. The potential – current density plots of the DL-EPR tests on type 316L stainless steel, with different degree of unidirectional rolling is shown in figure 3.4. As shown in the table 3.2 and figure 3.4, the DL-EPR ratio dropped slightly at 20% unidirectional reduction and then increased till 60% unidirectional reduction and finally dropped sharply at 80% unidirectional reduction.

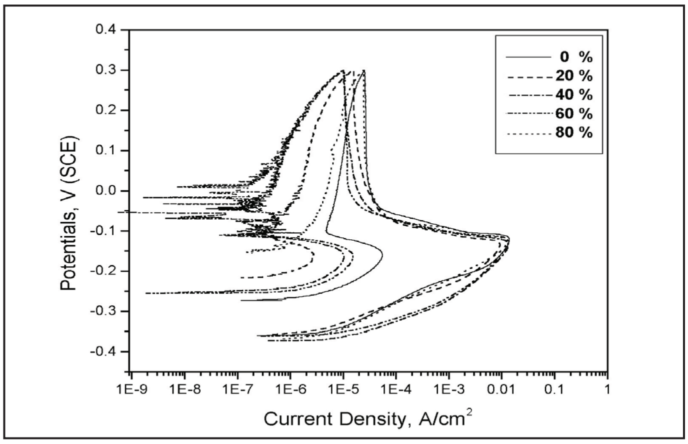

Fig. 3.4: The potential vs. current curve obtained in the DL-EPR test on the type 316L stainless steel. Unidirectional rolling being the mode of pre-solutionizing deformation, 0-80% reduction.

The SEM examination of the samples tested by DL-EPR and the samples tested as per practice B, A 262, ASTM [218] also show that that the IGC rates were low for the 80% cold rolled sample (B). The other samples, with 20-60% cold working (A), shows much higher corrosion rates. Figure 3.5(B) shows that high percentage of reduction (80% reduction with correspondingly very high concentration of random boundaries) did not result in any attack at the grain boundaries after the DL-EPR test.

Reduction Percentage	0%	20%	40%	60%	80%
Unidirectionally Rolled	0.4	0.0308	0.077	0.115	0.006

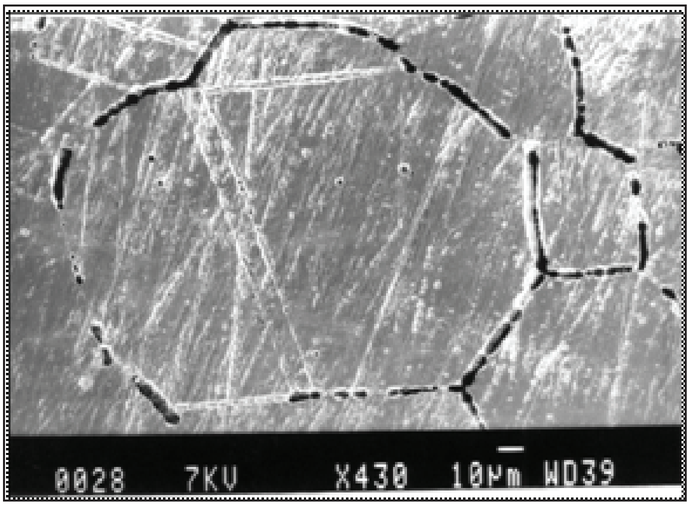

Fig. 3.5(A) Attack on grain boundaries at 60% unidirectional

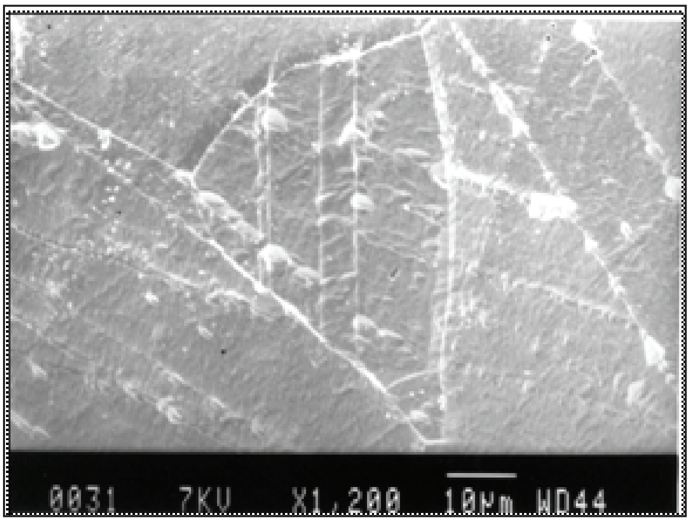

Fig. 3.5(B) No attack on grain boundaries at high percentage of reduction i.e. 80% unidirectional rolled, where grain boundary energy is high.

IV. Conclusion

- During deformation, there was a significant increase in copper and Sulphur.
- The DL-EPR test concludes that the ratio dropped slightly at 20% unidirectional reduction and then increased till 60% unidirectional reduction and finally dropped sharply at 80% unidirectional reduction as ahown in table 3.2 and fig.3.4.
- The SEM examination produced micrographs where the Intergranular Corrosion rates were low for 80% cold rolled sample.
- The other samples, with 20- 60% cold rolled, show much higher Intergranular Corrosion rates.
- The 80% cold rolled sample did not result in any attack as the grain boundary energy was high.

V. References

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