Precipitation Phenomena: Deformation and Aging

Edited by **B.N.Dey**

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B. N. Dey

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PREFACE

The articles included in this publication are from a program on precipitating alloys, mostly ferrous. These interesting investigations are of immense value for both nuclear and non-nuclear applications. The papers deal with the effects of aging and deformation, in theory and experiments. Because of time limitation, not all the papers intended for presentation at the conference, could be included in this publication. I am thankful to all the presenters, who have made this international conference possible. I am also grateful to all chairmen of the sessions for kindly agreeing to help for the smooth running of the program. I am thankful to all members of the sponsoring committee for their encouragements. I wish to thank also, my wife Gouri, my daughter Jhilmil and my son Spondon, for their patient assistance in collecting and reorganizing many sections of the edited manuscripts to fulfill our goal of the planned publication.

B. N. Dey

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PRECIPITATION PHENOMENA: DEFORMATION AND AGING-A PHENOMENAL AFFAIR

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Precipitation processes in alloys, both ferrous and non-ferrous, are extremely important for strengthening mechanisms in alloys and their structural applications in chemical, nuclear and non-nuclear industries, as well as other high technology areas.

Age-hardening alloys have been a subject of extensive studies in the past since Wilm (1911) first observed it in 1906 and Merica et al (1920) followed up with other investigations. About seven decades earlier, Karsten (1939) discovered one of the ingredients, viz. intermetallics, which are interrelated with the precipitation processes.

Precipitation may be defined as the decomposition of a solid solution (the parent matrix) into a new phase of different composition (the precipitate) and the initial solid solution with diminished solute concentration (depleted matrix). Changes in the physical properties (such as resistivity, etc.) chemical properties (such as corrosion, etc.) and the mechanical properties, such as hardness, damping and so on, accompany precipitation (see Christian, 1974; Smoluchowski, 1951; Ustinovhikov, 1938; Dey, 1965, 1965, 1986).
On aging some precipitating alloys after quenching from their solution treatment temperature, two processes usually occur before the equilibrium phases are finally formed:
i) Clustering: the process which, accompanied by a marked change in the electrical properties and by energy release-occurs at the very early stage of aging and results in the formation of a great number of sub-electronmicroscopic solute atom clusters;
ii) Zone formation: the process in which Guinier-Preston zones grow following initial clustering, and hardening as well as x-ray diffraction effects take place. The hardening effects has been attributed to the transition structures, which when transform to the equilibrium phases cause softening. Handling strain causing increase in the internal friction of furnace-cooled Al alloys testifies to this latter case (Dey, 1965).

Cold working, both prior to and after precipitation hardening treatments is a matter of intrinsic interest, as the changing concentration of imperfections affect both the precipitation and the reaction temperature. Physical, mechanical and other behaviors of the alloys are extremely sensitive to the deformation and subsequent processes.
Minor plastic deformation immediately after quenching increased the hardness of an Al alloy (Graf and Guinier 1954). Studies of different sorts involving carbide precipitations and effect of deformations have been reported during the last thirty years (Ustinovshikov, 1984, 1985). Also, continuous and cyclic deformation research with the help of modern day technology has shed new light into both the theory and the actual observations of ferrous and non-ferrous precipitating systems. The following papers will undoubtedly show the current state of the affair.

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EFFECTS OF PRIOR AND SIMULTANEOUS DEFORMATION ON THE SENSITIZATION OF AUSTENITIC STAINLESS STEELS

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ABSTRACT

The presence of prior or simultaneous deformation results in an acceleration in the kinetics and, perhaps, an alteration in the thermodynamics of sensitization. This has been observed in a study on the effects of prior and simultaneous strain on the precipitation of carbides and development of sensitization in Types 304 and 316 austenitic stainless steels. Samples were strained to different levels using standard and specifically designed variable strain tensile specimens. Carbide precipitation and sensitization behavior was examined using analytical electron microscopy (AEM) and electrochemical potentiokinetic reactivation (EPR) tests.

The extent to which the kinetics of sensitization is accelerated depends directly on the amount of strain present in the material. Specimens subjected to increasing strain levels sensitize at shorter times and achieve higher degree of sensitization (DOS) values. Lower chromium minimums and wider, asymmetric chromium-depletion profiles have also been observed (using AEM) in strained versus unstrained samples. Asymmetric depletion profiles are associated with selected grain boundaries where strain-induced grain boundary migration has taken place. Carbides on these grain boundaries tend to extend preferentially into one grain instead of growing as thin, flat plates as found on unstrained boundaries.

INTRODUCTION

Stainless steels (SS) are susceptible to intergranular (IG) corrosion and stress corrosion cracking (SCC) in certain aqueous environments (e.g., high-temperature oxygenated water). This susceptibility is caused by a change in the grain boundary microchemistry which occurs when the material is heat treated in, or slow cooled through, a particular temperature range (500-900°C).

The primary composition change involved is the depletion of chromium in the region adjacent to the grain boundary caused by grain boundary precipitation of chromium-rich carbides.[1-6] Chromium depletion (or sensitization) depends on a critical balance between the thermodynamics of carbide precipitation and kinetics of chromium diffusion. Thus, variables affecting this critical balance control the sensitization response of the material.

Plastic deformation has been known to be a key variable affecting rates of reactions occurring in materials. Reaction thermodynamics and kinetics, for example, may be modified by the deformation process.[7] Sensitization development is, likewise, altered by the presence of strain.[1,8-18] However, limited data exists to quantify the effects of straining on sensitization development. This data is vital to understanding the effects of thermomechanical processing and fabrication on the sensitization phenomena.

In this work, a systematic study has been carried out to evaluate the effect of straining prior to, or during, isothermal treatment on the sensitization response of Types 304 and 316 austenitic SS. Degree of sensitization has been measured using indirect electrochemical and direct analytical methods.

EXPERIMENTAL PROCEDURE

MATERIALS - High carbon, Types 304 and 316 stainless steel heats were used to examine the effects of deformation on sensitization. The composition of the heats are given in Table 1. All heats were received in the mill processed condition and solution annealed (1100°C/1 hour) before subsequent experimentation. Solution annealed specimen grain sizes were found to vary from 110 to 150μm.

DEFORMATION SPECIMENS - Three types of deformation tests were performed. They consisted of two types of prior deformation tests and one type of simultaneous deformation test. Prior deformation specimens were strained at ambient temperature prior to isothermal exposure while simultaneous deformation specimens were subjected to continuous deformation at a set strain rate during isothermal exposure.

One type of prior deformation specimens were given a selected prior strain and then subjected to an interrupted, additive isothermal heat treatment cycle. These specimens were fabricated from high-carbon Type 316 SS and were used to determine the effect of the magnitude of single-step prior strain on sensitization development. The second type of prior deformation specimens were fabricated from high-carbon Type 304 and 316 SS and were subjected to ambient straining at a fixed strain rate between each heat treating step of an interrupted, additive isothermal heat treatment cycle.

Strain magnitude effects of prior (single-step) deformation on sensitization were investigated using a specifically designed, variable strain tensile specimen. The specimen was flat, 300mm in length and had a width varying from approximately 25 to 7.5mm along a gage length of 175mm. Ambient uniaxial tensile testing on this specimen produced a range of strains from 0 to 40% prior to necking. Strains were estimated by marking the gage at 12.5mm intervals and measuring the reduction in cross sectional areas at each interval.

Sections corresponding to 0.4, 2.8, 8.5, 13.5, and 30% average strain were cut from the variable strain specimen. These were then aged alongside an unstrained, annealed specimen at 625°C. Incremental heat treatments were performed using variable time steps out to a total time of 100 hours at 625°C. Sensitization measurements were made on the material after each heat treatment step.

The effects of uniaxial deformation during isothermal treatment was investigated using flat, dogbone-shaped tensile specimens. Specimens were deformed at constant extension rates varying from 1 to 6% strain per hour. Isothermal heating was attained using a clamshell furnace surrounding the tensile specimens. Unstrained specimens were attached to the gage section of the tensile specimen during heat treatment to ensure that the control specimens were subjected to an identical thermal history. Temperatures were measured along the gage section during heat treatment and were found to be within 5°C of the specified 600°C for these tests. Initial isothermal exposure durations were between 0.5 and 1 hour at 600°C. Larger exposure times were subsequently used to achieve total test times between 5 and 15 hours. Degree of sensitization measurements were carried out after each isothermal exposure.

The multistep prior deformation test and control specimen configurations were the same as those used for the simultaneously strained specimens. The multistep specimens were deformed at ambient temperature at strain rates comparable to those used at elevated temperature for the simultaneously strained specimens. The magnitude of strain for each ambient strain cycle was similar to that received by the respective simultaneously strained specimen during its next thermal cycle. Therefore, the strain magnitude received by multistep prior and simultaneously strained specimens deformed at the same strain rate were similar at the end of each respective heating cycle. This was done to enable a more direct comparison between prior and simultaneous deformation. DOS measurements were made after each isothermal exposure step and prior to the next straining step.

SENSITIZATION MEASUREMENT - Sensitization response of the different specimens was measured using the EPR test. EPR tests were performed using the method proposed by Clarke.[19-21] The EPR test has

Table 1 - Bulk Compositions and Grain Sizes of Stainless Steel Heats

AISI	Heat	C	Cr	Ni	Mo	Mn	Si	P	S	N	B
304	C-6	0.062	18.48	8.75	0.20	1.72	0.39	0.013	0.013	0.065	---
	C-7	0.072	18.53	9.33	0.43	1.74	0.46	0.046	0.017	0.036	---
316	SS-16	0.058	17.11	11.43	2.16	1.77	0.41	0.014	0.005	0.008	0.002

been shown to be a reliable, quantitative method of measuring the DOS for a SS material and can be related to grain boundary chromium depletion.[5,6]

The laboratory variation of the EPR test was used to determine DOS for the single-step prior deformation specimens. The field variation of the EPR test was used to determine DOS for the multistep prior and simultaneous deformation tests. These test variations have been found to produce equivalent results.[22,23] Experimental parameters employed are summarized in Table 2.

Table 2 - Experimental Parameters for EPR Tests

Instrument:	Instruspec* Model WC-5
Electrolyte:	0.5 M H_2SO_4 + 0.01 M KSCN at 30°C
Specimen Preparation:	1-μm Diamond Polish
Passivation Conditions:	Potential of 0.2V (SCE) for 2 minutes
Reactivation Scan Rate:	6V/hour (AISI 304 SS) 3V/hour (AISI 316 SS)

*Registered Trademark

In addition to the indirect EPR method of detecting sensitization development, limited direct examination of changes in grain boundary microchemistry (using AEM) were also made. Samples were mechanically thinned (to less than 150μm) and subsequently jet polished using a solution of 3 volume percent perchloric acid in methanol. Examination of grain boundary microchemistry was made using a Philips EM 400T scanning transmission electron microscope (STEM) equipped with an ultra-thin window, energy dispersive x-ray detector. Quantification of x-ray spectra was made using the Cliff-Lorimer technique.[24]

RESULTS AND DISCUSSION

PRIOR DEFORMATION EFFECTS ON SENSITIZATION - The presence of strain prior to heat treatment sharply accelerated sensitization development. Specimens strained to levels greater than 1% were observed to sensitize more rapidly and achieve higher DOS values than unstrained specimens with identical heat treatment schedules (Fig 1). The DOS value after 100 hours of exposure was found to more than double due to the presence of 13.5% prior strain.

Prior deformation appears to linearly increase DOS, at least after 100 hours of exposure at 625°C and up to a strain level of 13.5% (Fig 2). Deforming to 30% strain level results in EPR-DOS values higher than that of the 13.5% strain sample. The data, however, has not been used for quantitative analysis due to the presence of intragranular attack.

The acceleration of sensitization with deformation illustrates the critical importance of thermomechanical, not just thermal, effects on sensitization development. An increase in the prestrain level is observed to decrease the "time to sensitize" the Type 316 heat (time to reach an EPR value of 5 C/cm^2) (Fig 3). This can be assumed to reflect a reduction in chromium carbide nucleation time as a function of strain present in the material.

The presence of strain prior to the sensitization heat treatment also has a pronounced effect on the rate at which the DOS value increases after initial sensitization. Acceleration of sensitization kinetics appears to be a direct function of the applied strain. Deformation induced accelerated sensitization development has also been observed by other investigators.[8-18]

Differences in sensitization kinetics with increasing deformation has been postulated to be due to increased dislocation density with strain, thereby enhancing dislocation pipe diffusion of chromium. If the effective chromium diffusivity is a function of mobile dislocations, then the density of dislocations created by the straining process will control chromium diffusion, which in turn controls carbide growth, depletion width and DOS. Hart has proposed a relation to account for changes in diffusivity of substitutional impurities with changes in dislocation density.[25] This relation has been quantified to account for dislocation density effects on chromium diffusivity in austenitic SS.[11]

A near-linear increase in dislocation density with strain has been observed by the authors after 100 hours of ageing at 625°C (Fig 4). The plot depicts a markedly higher dislocation density for the 13.5% strain specimen, as compared to the annealed (unstrained) specimen, and can explain the acceleration in kinetics of sensitization. Measurement of dislocation density values in as-strained specimens is being carried out to obtain a more fundamental under- standing of strain effects on chromium diffusivity and sensitization development in austenitic SS.

Optical micrographs (Fig 5) after EPR tests corroborate the changes in DOS found after 10 hours of exposure, as seen in Fig 6. The extent of grain boundary attack after 10 hours of ageing at 625°C was seen to increase with increasing strain level up to about 20% strain, while extensive intragranular attack is seen on specimens with strains greater than 20%. Observation of this nature have also been made in the work of Pednekar et al,[13] and other researchers.[13-17]

Increasing grain boundary attack as a function of strain suggests that deformation can promote a more uniform development of chromium depletion along grain boundaries in much shorter exposure times than thermal exposure alone. Continuous depletion along grain boundaries is often referred to as a prerequisite for IGSCC. The presence of EPR attack within the grains, noted at higher strain levels, may be beneficial in that it decreases available carbon for IG precipitation and may, therefore, decrease sensitization.

SIMULTANEOUS DEFORMATION EFFECTS ON SENSITIZATION - Simultaneous straining also dramatically accelerated sensitization development. Measurable DOS is observed in shorter times, and EPR-DOS reaches higher values during additive 600°C anneals in strained versus unstrained specimens. Sensitization response as a function of heat treatment time is documented in Fig 7 for the two Type 304 stainless steels (Heats C-6 and C-7) for a strain rate of 6%/hour. Large EPR-DOS values (>10 C/cm^2) can be noted after only 1 hour at 600°C in the deformed specimens, whereas more than 9 hours were required to approach a comparable EPR-DOS in the control specimens. EPR-DOS values of more than 35 C/cm^2 are observed after 9 hours for the C-6 strained specimen versus only about 5 C/cm^2 for the unstrained specimen.

Differences of this magnitude substantiate the critical importance of thermomechanical effects on sensitization development. A simple isothermal heat treatment of many hours at 600°C does not result in significant attack in the EPR test, only isolated regions along some grain boundaries are ditched. On the other hand, strained specimens show almost continuous grain boundary attack after only one hour at 600°C.

Additional simultaneous strain experiments were conducted on the C-6 SS to determine the effect of deformation rate on the sensitization development. Specimens were deformed at a rate of 1 and 3% strain per hour and compared to the 6% results. Sensitization kinetics scaled consistently with increasing deformation rate, as shown in Fig 8. After 9 hours, EPR-DOS is about 7, 15, 23, and 35 for deformation rates of 0, 1, 3, and 6% per hour. Data for all four isothermal control (unstrained) specimens are also plotted in Fig 8 to give some indication of the data scatter in these measurements. Decreasing "time to sensitize" (EPR = 5 C/cm^2) observed with increasing strain rate (Fig 9) also indicate a decrease in nucleation time with increasing deformation. These differences in kinetics with increasing deformation rate can be explained by changes in chromium diffusivity caused by enhanced dislocation pipe diffusion of chromium.

Grain boundary carbide precipitation and chromium depletion characteristics were examined on strained and unstrained Type 304 SS specimens. Bright field TEM micrographs are shown in the top of Fig 10 for the C-7 specimen after 9 hours at 600°C. EPR results for this specimen were presented in Fig 7a. A deformation rate of 6% per hour produces an accumulated strain of more than 50%. This damage is reflected in the high dislocation density which can be seen in the bright-field image (upper right, Fig 10). Dislocations are continuously created with time during the simultaneous deformation process leading to a continuously increasing dislocation density during isothermal exposure.

Grain boundary carbide morphologies are also quite different between the strained and unstrained samples. Carbides are elongated along the boundaries in the isothermal case, but extend preferentially into one grain with deformation. Not all boundaries exhibit this appearance. It is probable that only interfaces which are properly oriented with the applied uniaxial stress are influenced. Deformation may promote discontinuous carbide growth due to short-circuit diffusion paths created by the influx of dislocations. Discontinuous or cellular precipitation involves grain boundary diffusion and migration. Betrabet et al.[26] have documented discontinuous precipitation of $Cr_{23}C_6$ carbides in Type 304 SS after isothermal heat treatments.

Discontinuous carbide precipitation mechanisms, whether interface-energy driven[27,28] or chemically driven[29] will lead to the formation of asymmetric chromium depletion profiles. Precipitation resulting from volume diffusion where grain boundary migration does not occur will create nearly symmetric profiles.

Chromium depletion was mapped for both strained and unstrained specimens. Consistent with its much larger EPR-DOS, the strained specimens exhibited lower chromium minimums and wider depletion widths. Depletion profile characteristics for the two C-7 specimens are illustrated in the lower portion of Fig 10.

Differences in symmetry between the two profiles can be noted with the strained specimen's profile skewed to one side of the interface. Depletion extends preferentially into the same grain as noted for carbide growth. Chromium minimums were measured at about 8 wt% for the strained versus 13 wt% for the unstrained specimen. The measured minimum for the deformed SS at

600°C is about 2 wt% lower than that observed for an isothermally treated specimen at 600°C, regardless of heat treatment time.[5]

The asymmetry in depletion profiles of strained specimens typically results in the minimum chromium concentration being measured some distance from the grain boundary. Minimum chromium was found to be about 20 to 30 nm from the interface as demonstrated for the C-7 specimen in Fig 10. Grain boundary migration appears to be a key in the accelerated sensitization kinetics. It should be noted that asymmetric chromium depletion profiles caused by grain boundary migration are also possible in isothermal, unstrained specimens. Their development is, however, after significantly longer ageing times, and the presence of strain appears to be a key factor in accelerating their occurrence. Deformation promotes grain boundary migration at lower temperatures and directly influences chromium depletion width and DOS. The effect on chromium minimum concentration due to deformation may be caused by changes in grain boundary or carbide interface thermodynamics.

Simultaneous deformation was also found to accelerate sensitization development in the Type 316 SS at 600°C. Carbide nucleation and subsequent sensitization are slow at 600°C due to the presence of molybdenum in these heats. Data for unstrained and specimens strained at 6%/hour are presented in Fig 11. Without deformation, measurable EPR-DOS is not detected until an exposure of 30 hours and only reaches a value slightly more than 5 C/cm^2 after 50 hours at 600°C. The simultaneously deformed specimen, on the other hand, shows a measurable DOS at short times (approximately 2 hours) and increases to about 10 C/cm^2 after 5 hours, 20 C/cm^2 after 10 hours and 25 C/cm^2 after 13 hours. These values are significantly larger than the control specimen reached in 50 hours and indicate changes in both "time to sensitize" and kinetics of chromium depletion zone development. Simultaneous deformation effects are consistent for both Type 304 and 316 SS.

MULTISTEP PRIOR DEFORMATION EFFECTS ON SENSITIZATION - In order to better compare simultaneous and prior deformation, multistep prior deformation experiments were performed on both Types 304 and 316 SS. Specimens were subjected to strain magnitudes, strain rates and isothermal time steps identical to simultaneously strained specimens, except that the deformation was carried out at ambient temperature. Multistep prior deformation EPR-DOS tracked quite well with simultaneous strain data as shown in Figs 12a and b. For the Type 316 case (Fig 12a), the prior deformation specimens exhibited measurable DOS after only 1 hour versus 3 hours for the simultaneous strain and about 30 hours for the unstrained specimen. It appears that the simultane- ously strained specimen does tend to promote slightly larger EPR-DOS as time at temperature is increased. The difference is, however, not thought significant. In fact, the opposite behavior, when comparing multistep prior and simultaneous strain, is demonstrated in Fig 12b for the Type 304 heat. Prior deformation rates of 3% strain per hour resulted in larger EPR-DOS at all times. Detailed conclusions cannot be made from these few comparisons, but do indicate that prior or simultaneous deformation promote similar acceleration of sensitization kinetics. This may imply that changes in density of dislocations due to simultaneous straining during heat treatment are similar to that produced during prior deformation and subsequent isothermal treatment.

Chromium depletion characteristics were also examined for the Type 304 heat specimens subjected to multistep prior deformation after 9 hours at 600°C. Minimums were again lower than for the unstrained isothermal exposures. However, minimums were still higher than for the simultaneous deformed specimens. Chromium concentrations were measured down to about 9.2 wt% for the prior deformation specimens versus 8 wt% for the simultaneous specimens. Profiles again were found to be slightly asymmetric after the cumulative prior deformation and heat treatment sequence. Total depletion zone widths were comparable for the two deformation conditions. It is not known whether differences in profile characteristics are significant, thereby implying mechanistic differences in the effects of prior and simultaneous deformation on sensitization, or whether data scatter masks the fact that they are essentially the same.

SUMMARY

The effects of prior and simultaneous deformation on sensitization development have been studied for Types 304 and 316 austenitic SS. The experimentation discussed above indicates deformation is a key variable affecting the sensitization response of both 304 and 316 SS.

Lower grain boundary chromium minimums are observed in deformed specimens, which may indicate an alteration in reaction thermodynamics with deformation. Results also indicate an acceleration in sensitization kinetics with straining prior to, or simultaneous with, the sensitization heat treatment. Specimens subjected to increasing deformation exhibit lower "times to sensitize" and achieve higher DOS values as a function of both strain and strain by an increase in chromium diffusivity which, in turn, is hypothesized to be due to a deformation induced dislocation density increase. Wider, asymmetric chromium depletion profiles observed on strained specimens may also contribute to the observed increase in kinetics of sensitization development.

Deformation also promotes a higher density of grain boundary carbide precipitates, different carbide morphologies and a decrease in the time needed to achieve a uniform chromium depletion zone along grain boundaries. The latter phenomenon has been associated with material susceptibility to intergranular corrosion and stress corrosion. Deformation above 20% prior strain, however, induces a combination of inter- and intra-grain precipitation and, therefore, reduces sensitization susceptibility.

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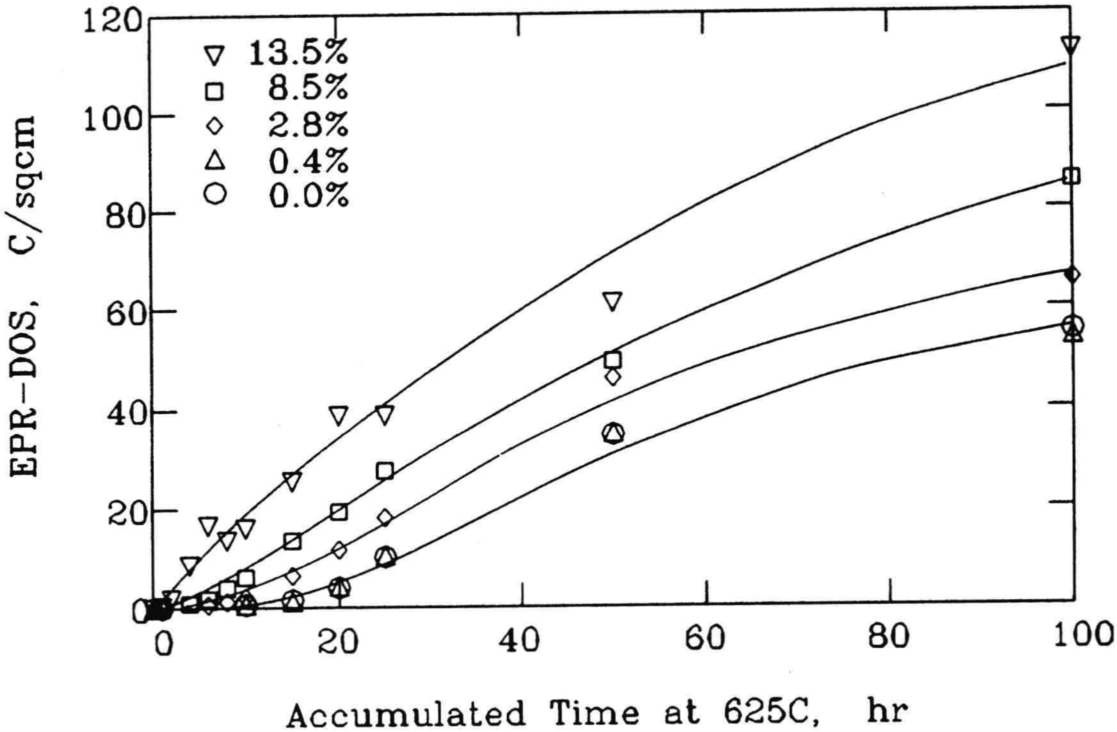


Fig. 1 - Prior Deformation Effects on Sensitization Development in Type 316 SS.

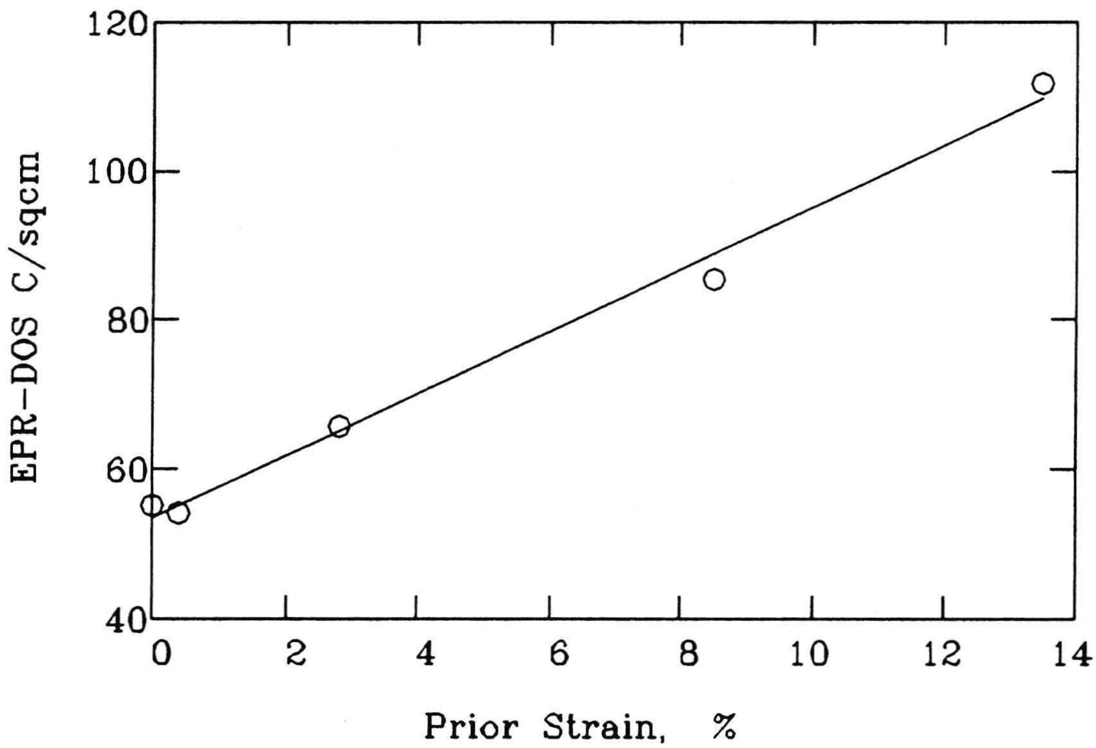


Fig. 2 - Variation of EPR-DOS with Prior Strain After 100 Hours of Ageing at 625°C for Type 316 SS.

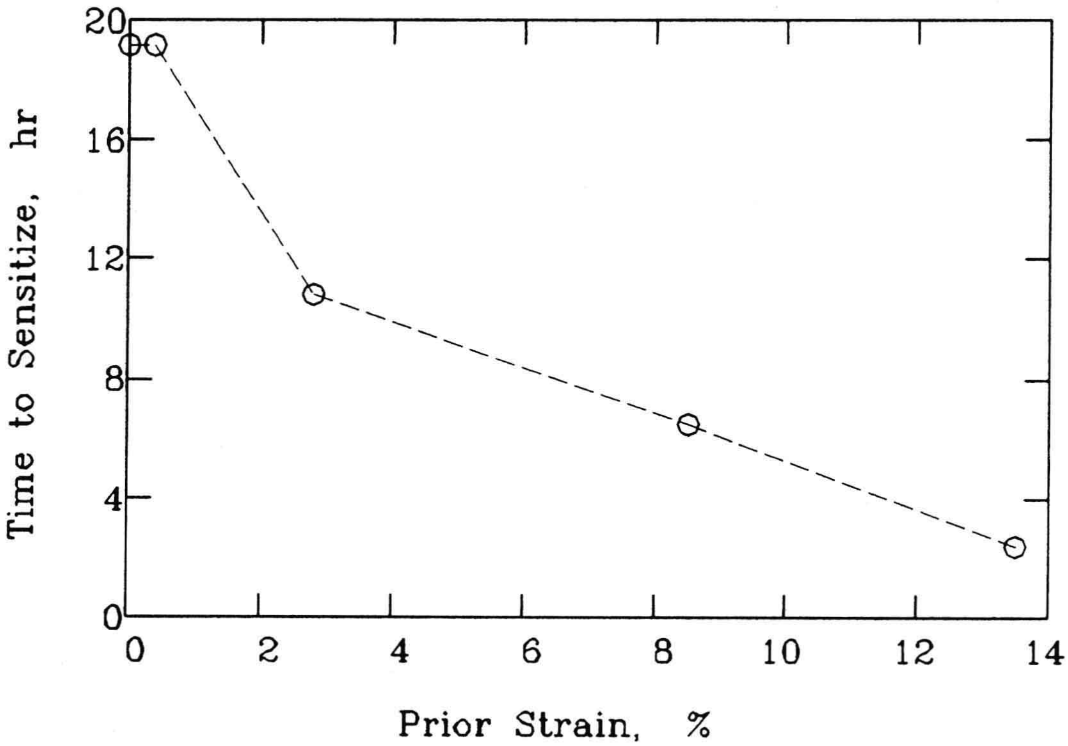


Fig. 3 - Prior Strain Effects on "Time to Sensitization" (EPR-DOS = 5 C/cm^2) for Type 316 SS.

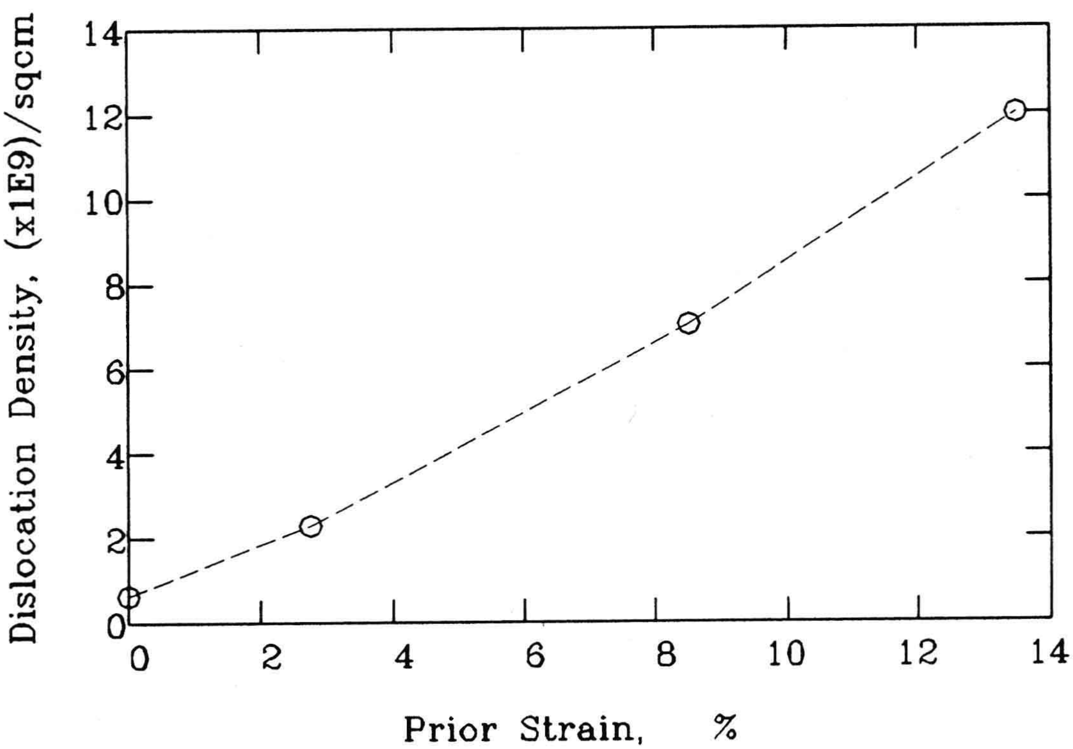
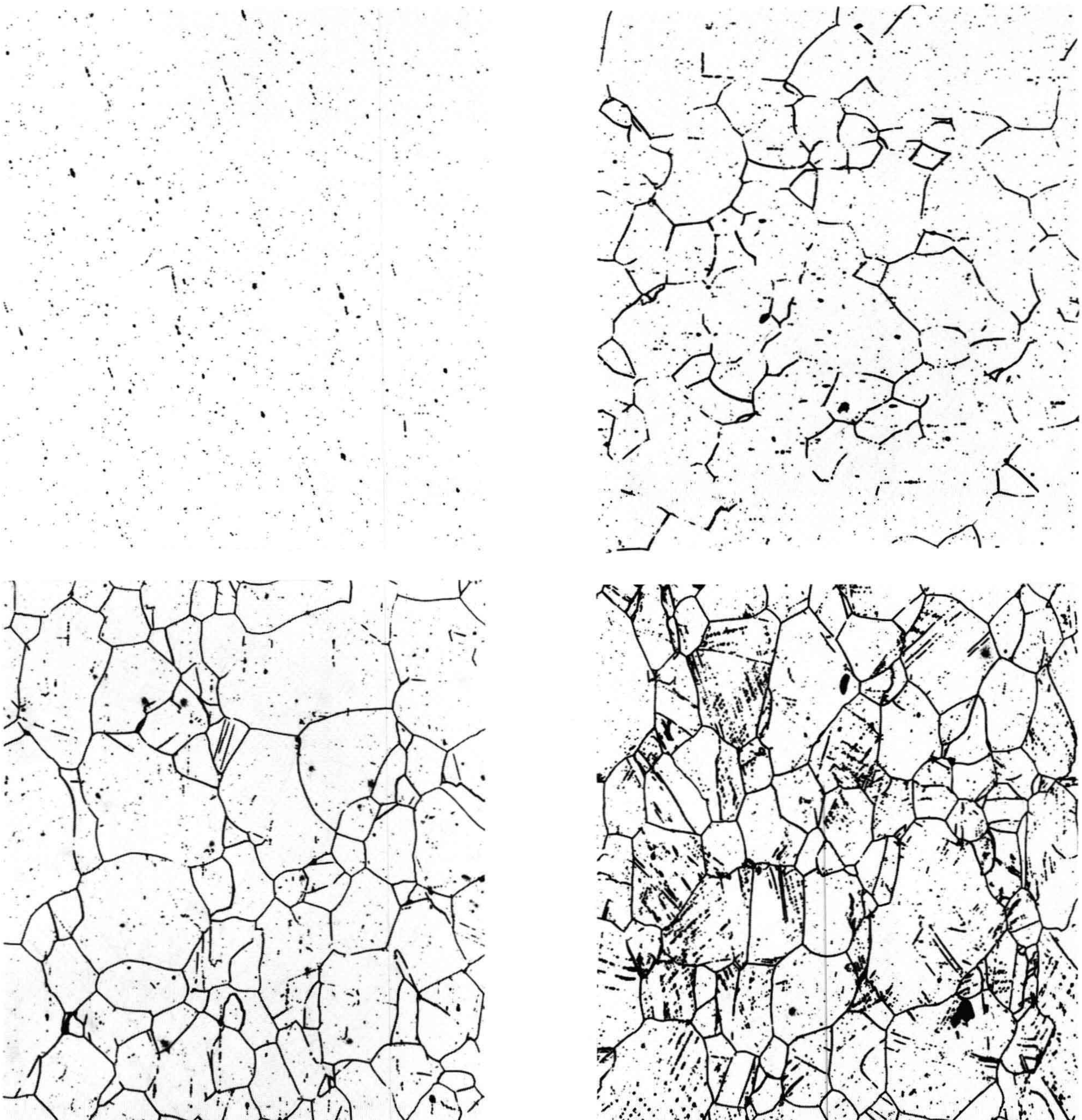


Fig. 4 - Variation in Dislocation Density with Prior Strain for Type 316 SS. Dislocation density measurements were made on prior strained specimens after 100 hours of ageing at 625°C.



(c) (d)

Fig. 5 - Optical Micrographs Indicating Changes in Extent of EPR Test Attack for (a) 0%, (b) 8.5%, (c) 13.5%, and (d) 30% Prior Strained Specimens (Type 316 SS).