

## TRANSFORMATIONS INDUCED IN AUSTENITIC STAINLESS STEELS BY SHOT PEENING

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### ABSTRACT

*The presence of martensite in a nominally austenitic stainless steel can have a profound effect on the steel's physical, chemical, mechanical and service properties. Plastic deformation can induce transformation of austenite to martensite. AISI 304 and 316 steels were peened using a broad range of peening severities. Peened and unpeened specimens were examined using 'direct comparison' XRD, 'two-exposure' XRD, hardness surveys, metallography and qualitative corrosion testing. The 304 grade was found to contain approximately 50% of martensite after peening with high levels of compressive residual stress in both austenite and martensite phases. No transformation was observed in the 316 grade. Hardening by peening occurred to similar depths in both grades but the degree of surface hardening was much higher in the 304 grade (750HV cf 550HV). Corrosion tests showed that the two-phase, peened, 304 grade had a substantially reduced corrosion resistance as compared with the single-phase, peened, 316 grade.*

### KEY WORDS

Austenite, martensite, steel, stainless, stress, corrosion, hardening, transformation, peening.

### INTRODUCTION

The maximum temperature at which plastic deformation is capable of transforming metastable austenite into martensite is known as the  $M_d$  temperature. This transformation is caused by the strain energy supplied by the deformation of the steel. The  $M_d$  temperature of any given steel will always be higher than the corresponding  $M_s$  temperature. That  $M_s$  temperature must be below room temperature for a steel to be fully-austenitic at room temperature on cooling. If, however, the  $M_s$  temperature is close to room temperature then the  $M_d$  temperature will be higher than room temperature. In that case the steel will be susceptible to martensitic transformation by plastic deformation at room temperature (R.T.) see Fig.1.

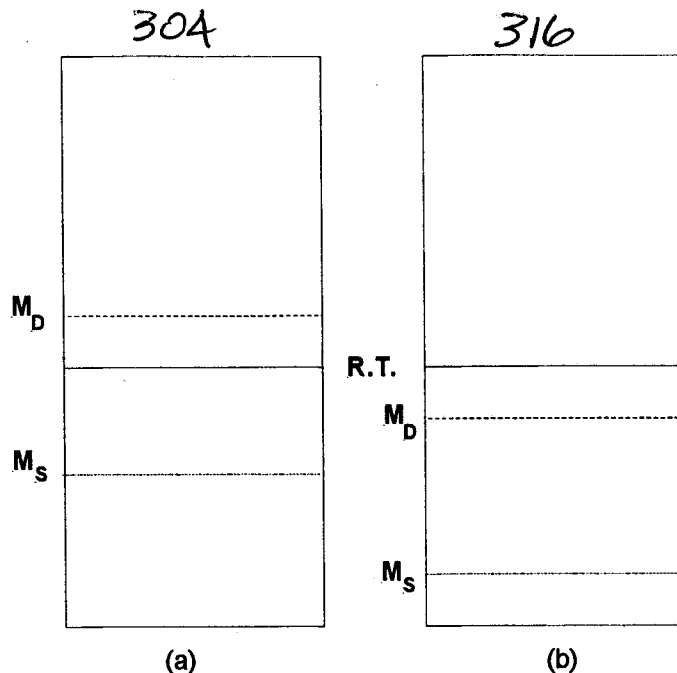


Fig.1 Schematic representation of relationships between critical temperatures for transformable steel, (a), compared with stable steel, (b).

If an austenitic steel is transformed to a two-phase martensite/austenite structure most of its physical, chemical and mechanical properties will be affected. The body-centred-tetragonal martensitic phase is ferromagnetic whereas the close-packed face-centred-cubic austenite phase is non-magnetic. Martensite is not 'close-packed' so that dimensional stability is affected. In essence the same mass of steel occupies a larger volume as martensite than it does as austenite. Corrosion resistance will normally be much lower for a two-phase material than it is for a single-phase material of similar chemical composition. The U.T.S. and hardness will be increased whereas ductility and toughness will be reduced for the two-phase condition. Fatigue performance will also be affected.

The susceptibility to transformation is a function of the composition of the steel. Several empirical relationships between the  $M_s$  temperature and the composition of the steel have been published<sup>1-4</sup>. These allow estimates to be made of the  $M_s$  temperature for steel of a known chemical composition. An empirical relationship has also been published<sup>2-4</sup> for estimating the  $M_D$  temperature for a particular combination of applied strain and percentage of induced martensite in a steel of known chemical composition. It should be noted that the  $M_D$  is a function of strain and percentage transformation. The greater the degree of strain the higher will be the  $M_D$  temperature. Shot peening has been selected for this research because it imparts a very large amount of surface plastic deformation. This extensive deformation should maximise transformation in any susceptible austenitic steel and, conversely, demonstrate resistance to transformation in a non-susceptible steel. The available peening facility is capable of controlling the amount of deformation imparted. A primary function of shot peening is to induce a surface layer containing beneficial compressive residual stress. X-ray diffractometry can therefore be used to study not only the amount of martensite formation but also the types of residual stress developed in the separate martensite and austenite phases.

Several studies have been published indicating the significance of martensite formation in austenitic stainless steels, e.g. Kirk<sup>5</sup>. Iida and Tosha<sup>6</sup> carried out a variety of investigations

into transformation induced in SAE 304 grade stainless steel. They found that substantial transformation occurred and that high levels of residual stress, presumably in the austenite phase, were induced by shot peening.

## **AIMS AND OBJECTIVES**

- To determine which compositions of austenitic stainless steel will transform upon being plastically deformed, i.e. steels that have an  $M_s$  below room temperature but an  $M_d$  temperature above room temperature.
- To determine the type and level of residual stress in both the austenitic and martensitic phases of a transformed stainless steel.
- To ascertain the percentages of martensite and austenite phases within the transformed steel by application of a 'direct comparison' X-ray diffractometer technique.
- To investigate the effect of peening intensity on the transformation characteristics of an austenitic stainless steel found to transform to martensite.
- To demonstrate, qualitatively, the change in corrosion resistance for a two-phase, transformed steel.

## **EXPERIMENTAL METHOD**

### **Material Selection**

The composition of a suitable austenitic stainless steel, which would display martensitic transformation, was not initially known. The published empirical equations were not able to predict appropriate compositions. It was therefore decided to carry out direct testing for transformability. These simple tests involved mechanical deformation (bending and hammering) of a range of available sheet stainless steel compositions, guided by the empirical equations. Any transformation that had occurred in the steel would be indicated by the initiation of ferromagnetism. This could be detected very simply either with the aid of a compass or by suspending the steel with a thread and attempting to attract the steel from its central position with a magnet. X-ray fluorescence spectrometry was then conducted on appropriate specimens to determine their chemical composition. On the basis of these tests grades 304 and 316 were selected as representing susceptible and non-susceptible sheet steels respectively.

### **Plastic deformation**

As the precise degree of plastic deformation that was required to transform the austenitic steel partly to martensite was not known, a broad range of shot peening treatments was used. To achieve this range of treatments sheet specimens were peened at 4, 5, 6 and 7 bar pressure, at a distance of 181 mm from the nozzle, for 30 seconds using S170 shot. During peening the specimens were held by a standard Almen strip holder having previously been cut to the standard Almen strip size.

### **X-ray Residual Stress Analysis**

This technique allows the separate residual stresses in different phases to be determined rather than the overall value indicated by deflection methods<sup>7</sup>. The residual stress within both the austenite and martensite was assessed. This was achieved using the standard

diffractometer method<sup>8</sup>, often referred to as the 'two-exposure method'. In this technique two measurements of the strain, as indicated by the inter-planar spacing of the diffracting planes (hkl), are taken at an angle  $\psi$  to each other. As the stress perpendicular to a surface is zero, only two strain measurements are needed to determine both the direction and the magnitude of the stress causing the measured strains. It has been shown that the residual stress at the surface,  $\sigma$ , is:

$$\sigma = \frac{E \cot \theta (2\theta_0 - 2\theta_\psi)}{2(1 + \nu) \sin^2 \psi} \quad (1)$$

Where  $2\theta_0$  = The position of the peak at  $\psi = 0^\circ$   
 $2\theta_\psi$  = The position of the peak at  $\psi = \psi^\circ$   
 $E$  = Young's Modulus  
 $\nu$  = Poisson's Ratio

Two-exposure measurements were made for both the austenite and martensite phases using chromium  $K_\alpha$  radiation and examining the 200 and 211 planes respectively.

### Direct Comparison XRD Technique

This technique<sup>9</sup> was used to quantify the relative amounts of each phase – martensite,  $\alpha'$ , and austenite,  $\gamma$ . Quantitative analysis of this nature is affected by the fundamental difference in the crystallographic structure of the two phases, austenite being f.c.c. and martensite being b.c.t. Account has also to be taken of the several factors in the following equation, which relates the measured integrated intensities of the austenite and martensite diffraction peaks,  $I^{hkl}$ , to their relative volume fraction,  $V$ .

$$\therefore \frac{V_\gamma}{V_\alpha} = \frac{I_\gamma^{hkl} \left( \frac{1}{v_\alpha^2} \right) [ |FF|^2 p LP e^{-2M} ]_\alpha}{I_\alpha^{hkl} \left( \frac{1}{v_\gamma^2} \right) [ |FF|^2 p LP e^{-2M} ]_\gamma} \quad (2)$$

Where,  $V_\gamma$  and  $V_\alpha$  = Volume fraction of the  $\gamma$  and  $\alpha$ -phases respectively,  $|FF|$  = structure factor times its complex conjugate,  $p$  = multiplicity factor,  $LP$  = Lorentz polarisation factor,  $e^{-2M}$  = Debye-Waller temperature factor and  $v$  = volume of the unit cell.

### Metallographic Studies

Various metallographic studies of the steels were conducted including a simple 4% sodium chloride solution corrosion test, standard microhardness surveys and general optical and SEM microscopy.

## EXPERIMENTAL RESULTS

Initial work to identify a material suitable for studying the martensitic transformation indicated that a steel having a composition of approximately 18Cr/8Ni transformed readily when plastically deformed. X-ray fluorescence spectrometry was used to find the exact composition of the steel. This gave a composition: Ni-8.6%, Cr-17.7%, Mo-0.1% and Mn-1.6%. A

commercially-available steel of similar composition was AISI 304, this steel having a composition of: C-0.08%max, Ni-8/10%, Cr-18/20%, Mo-0.6%max and Mn-2.0%max.

A steel that did not transform when plastically deformed was also to be used so that the differences between a transformed and an untransformed steel could be assessed. The best estimate for an appropriate, available, grade of stainless steel was AISI 316 which has the composition: C-0.08%max, Ni-10/14%, Cr-16/18%, Mo-2/3% and Mn-2.0%max. The higher nickel content of this steel is a primary reason for the greater stability of the austenite phase.

Calculations of the  $M_s$  and  $M_d$  temperatures for 304 and 316 grades were carried out using the following equations:

$$M_s(^{\circ}\text{C}) = 502 - 810(\% \text{C}) - 1230(\% \text{N}) - 13(\% \text{Mn}) - 30(\% \text{Cr}) - 12(\% \text{Ni}) - 54(\% \text{Cu}) - 46(5\text{Mo}) \quad (3)$$

$$M_d(^{\circ}\text{C}) = 413 - 462(\% \text{C} + \text{N}) - 9.2(\% \text{Si}) - 8.1(\% \text{Mn}) - 13.7(\% \text{Cr}) - 9.5(\% \text{Ni}) - 18.5(\% \text{Mo}) \quad (4)$$

\* - this refers to a 30% true strain inducing 50% of martensite in austenite. The values obtained by substitution in the equations are shown in Table 1.

**Table 1 Typical compositions, in percentages, for 304 and 316 grades of stainless steel and calculated  $M_s$  and  $M_d$  values using equations (3) and (4).**

Grade	C	Ni	Cr	Mn	Si	Mo	C + N	$M_s - ^{\circ}\text{C}$	$M_d - ^{\circ}\text{C}$
304	0.08	10.5	20	2	1	0.6	0.08	-171.4	-34.21
316	0.08	14	18	2	1	3	0.08	-362.8	-84.46

It should be noted that equations (3) and (4) are strictly empirical and do not take account of interactions between alloying elements. The calculated values show that 304 grade is predicted to be much less stable than is 316 grade. A temperature of  $-362.8^{\circ}\text{C}$  is clearly impossible but serves to indicate that cooling to absolute zero would not induce thermal transformation. Grade 304 had an estimated  $M_d$  temperature well below room temperature but that is for only 30% true strain. The predicted temperature would be much higher for very large plastic strains.

After peening, using the previously stated parameters, the residual stress in the 304 steel was determined using the standard 'two-exposure' diffractometer method. The data can be seen in Table 2.

**Table 2. Surface Residual Stress values for AISI 304 Steel Peened at Different Pressures.**

Peening Pressure - bar	Residual stress - MPa	
	Martensite phase	Austenite phase
4	-859	-825
5	-714	-522
6	-610	-516
7	-634	-247

The trends in the data are (i) that the value of residual stress in both phases seems to reduce with increasing peening pressure and (ii) that the level of residual stress in the martensitic phase is higher than that of the austenitic phase.

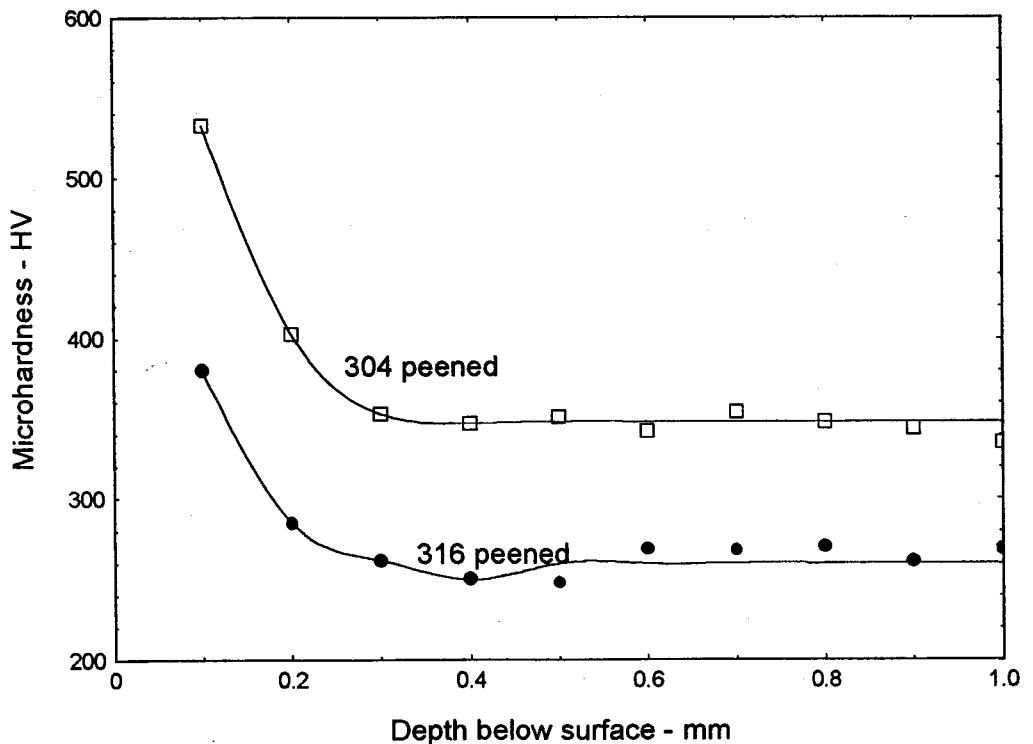
The measured volume fractions of martensite and austenite in AISI 304 steel specimens peened at different pressures are given in Table 3.

**Table 3. Volume Fractions of Martensite and Austenite in AISI 304 Steel Peened at Different Pressures.**

Peening Pressure – bar	Volume fraction	
	Martensite phase	Austenite phase
4	0.48	0.52
5	0.54	0.46
6	0.47	0.53
7	0.49	0.51

The data indicates that there is no significant effect of peening pressure on austenite transformation. Almost 50% transformation to martensite was achieved even with the lowest peening pressure.

The data from the microhardness surveys is given in Fig.2. It can be seen that the peening treatment has caused a hardening of the surface in both the transformed 304 and the untransformed 316 grades. There is, however, a much greater degree of hardening in the 304 due to the formation of the very hard martensite phase. Extreme surface hardness values are not included since the measurements were made on mounted cross-section specimens.



**Fig.2 Microhardness surveys of shot peened layers for 304 and 316 grade stainless steels.**

The outcome of the corrosion tests was that after one week in a 4% NaCl solution there were visible signs of corrosion on a peened and hence partially transformed AISI 304 specimen. This corrosion appeared as typical iron oxide rust-coloured pits on the peened surface with streaks of corrosion product along the cut edges. The unpeened AISI 304, peened 316 and unpeened 316 specimens showed no visible signs of corrosion.

SEM microscopy showed that the 304 surface was much less textured by the peening treatment when compared with the 316, see Fig. 3. This is again presumably due to the hard, less deformable, martensite formed at the surface of the 304.

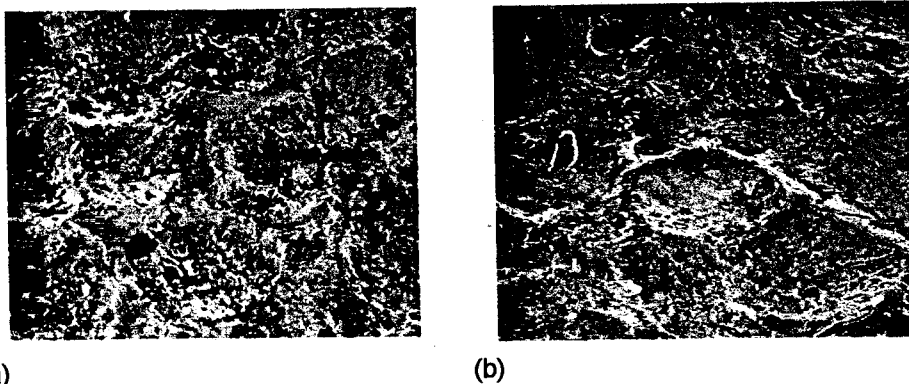


Fig.3 SEM scans of 304, (a), and 316, (b), steels peened at 7 bar.

## DISCUSSION

The observation that compressive residual stresses are present in both phases of the peened 304 steel is very important for fatigue resistance. The higher level of stress in the martensite phase is due to its having a much higher yield point than that of the austenite phase. It was curious, however, that the residual stress was lower in more severely peened material. This is, presumably, due to the phenomenon known as "work softening". During peening work-hardening of the surface rapidly reaches a maximum. Multiple impacting of a given area can thereafter induce work softening and attendant reduction in residual stress level.

The two grades of stainless steel used, 304 and 316, are commonly used commercial grades. It is, therefore, very significant that one grade has approximately 50% of martensite after peening and the other none. The technique used for the determination of the volume fraction of the phases present in each specimen, namely XRD direct comparison, worked very well. A common source of error was avoided by calculating the several parameters in the direct comparison equation specifically for the two different steels. For shopfloor estimation simple magnetic tests will provide a qualitative indication of martensite formation.

The other physical tests, i.e. hardness surveys and corrosion tests, displayed predictable changes in properties due to the martensite formation, i.e. increased corrosion susceptibility and greater hardening (above that simply due to peening). It is apparent that the observed changes in properties are due to the martensite formation and not only to the peening treatment as they are over and above that of the variations exhibited in the AISI 316.

The observed differences in behaviour of the 304 and 316 stainless steels are essentially caused by differences in the chemical composition. The 316 stainless steel has up to 4% more

nickel and about 2% more molybdenum content. Both molybdenum and nickel are austenite stabilisers, resulting in the inherent stability of the austenite of 316 at room temperature.

## CONCLUSIONS

1. Martensite formation was easily induced by plastic deformation in the AISI 304 stainless steel whilst no martensitic transformation could be engendered, even with gross surface plastic deformation, in the AISI 316 stainless steel.
2. Approximately 50% transformation of austenite to martensite was observed for all severities of shot peening AISI 304 grade steel..
3. The increase in both the hardness and the corrosion rate of the transformed 304 grade steel was due to the formation of martensite.
4. Transformation caused the 304 material to become magnetic due to the ferromagnetic attributes of the martensitic phase.
5. The additional nickel and molybdenum in AISI 316 grade steel imparts greater austenitic stability, such that extensive plastic deformation at room temperature leaves the austenite untransformed.
6. Substantial levels of surface compressive residual stress were observed in both the martensite and austenite phases of the peened 304 grade stainless steel.

## FURTHER WORK

It would be of interest to quantify the relationship between amount of transformation and the degree of plastic deformation for the 304 grade of austenitic stainless steel. This could be based on using either tensile test or bend specimens that would allow controlled amounts of plastic deformation to be applied. The measurement of progressive residual stress development would be possible using bend specimens – due to the inhomogeneous plastic deformation involved. A wider range of austenitic stainless steels grades could also be examined. Work could be done on quantifying the magnetic properties of martensite in order that a quicker and simpler shop-floor determination of the level of induced martensite could be established. Studies could also be carried out to quantify the differences in corrosion behaviour of two-phase and single-phase stainless steels. This should involve a wider range of corrosion conditions than simply one sodium chloride solution at room temperature.

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