Effect of Plastic Deformation on the Magnetic Properties 304 Stainless Steel During Tensile Loading

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Abstract - The present investigation addresses effect of tensile deformation on the magnetic properties of virgin 304SS as well as cold rolled samples containing a low volume fraction of 12% and 17% martensite. In-situ Non-destructive evaluation (NDE) techniques by magnetic Barkhausen emission (MBE) and magnetic hysteresis loop (MHL) measurement were used for evaluation of plastic deformation during tensile loading. Both the techniques indicated different stages of variation in magnetic properties with progressive plastic deformation. The trend of coercivity and Barkhausen measurements also throw light on the ductile and brittle fracture occurring in virgin and cold worked samples with validation using SEM fractography.

1.0 Introduction

The 304 stainless steels are extensively used in nuclear power plants and in many structural applications. When this austenitic steel undergoes plastic deformation it transforms from its non-magnetic cubic closed $\gamma$-phase to a bcc $\alpha$-martensite phase which is ferromagnetic [1,2] through a non-ferromagnetic $\epsilon$-phase. Due to such a transition into ferromagnetic state, magnetic techniques are effective tools to monitor the effects of the deformation behaviour. The prominent techniques include magnetic hysteresis loop (MHL) and magnetic Barkhausen emissions (MBE) measurement. Most of the reports refer to the response of these evaluations in terms of the effects of martensite content in 304SS with progressive cold rolling [3,4]. However, it is a fact that many of the structures experience unidirectional tensile loading which incur lots of defect formation like stresses, voids etc along with such deformation induced martensite generation and growth. The formation of defects and magnetic phases give complex type of output signals, the analysis of which is essential for assessment of structural integrity of component subjected to tensile deformation.

The present investigation was aimed at in-situ nondestructive magnetic evaluation of 304 stainless steel during tensile deformation. The study has been carried out on its virgin sample which is non-magnetic and also on cold rolled materials containing low volume fraction of $\alpha$-martensite which is ferromagnetic.

2.0 Experimental

Flat tensile specimens were prepared from virgin 304SS and cold-rolled sheets. The 35% and 40% cold rolled samples contained 12% and 17% of martensite respectively as measured using a stress analyser of x-ray diffractometer [Siemens D-500]. The virgin and
cold rolled materials were designated as sample #V, 12M and 17M were subjected to a continuous tensile loading in an Instron machine. Magnetic hysteresis loops (MHL) were obtained using a computer controlled hysteresis loop tracer at a quasi-dc field of 50mHz. The Magnetic Barkhausen emission (MBE) study was carried out at 40Hz frequency using surface probe. The MBE spectrum was analysed using a band pass filter of frequency of 30kHz-300kHz. The fractography was observed using Scanning electron microscope, SEM (Jeol-400).

3.0 Results and discussion

Fig-1 shows the optical micrographs of virgin and cold rolled samples. The cold rolled samples show martensite formation at shear bands whose percentage was 12% for 35% cold worked and 17% for 40% cold worked as determined from XRD- study. The samples were subjected to tensile loading till fracture and their tensile test curves are show in fig-2. The results of tensile deformation on the 304SS virgin as well as cold worked samples are shown in table-1. The YS and the ultimate tensile strength (UTS) increased while the percentage elongation decreased with martensite content.

<table>
<thead>
<tr>
<th>Virgin / % Cold rolled</th>
<th>Initial Martensite content (%)</th>
<th>Yield Strength YS (MPa)</th>
<th>Ultimate Tensile Strength (UTS)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Virgin</td>
<td>-</td>
<td>270</td>
<td>590</td>
<td>9.76</td>
</tr>
<tr>
<td>30%</td>
<td>12</td>
<td>725</td>
<td>981</td>
<td>8.00</td>
</tr>
<tr>
<td>40%</td>
<td>17</td>
<td>810</td>
<td>1073</td>
<td>4.85</td>
</tr>
</tbody>
</table>

With progressive tensile loading, the virgin and cold worked samples exhibited significant variation in magnetic hysteresis properties and Barkhausen emissions (MBE). activity as shown in fig-3 and fig-4 respectively. At low plastic deformation of the virgin sample (upto 1.3% strain, stage-I) the coercivity increased with strain indicating the magnetic hardness. At this stage martensite started forming randomly and these martensites were unable to interact with each other magnetically due to large separation. At this stage the coercivity increased with the increase in martensite grain size [5]. However, when the martensite grains grew large enough, the grains came closer and coupled ferromagnetically with each other. At this stage (Stage-II) the coercivity started decreasing. This stage extended from 1.3% to 2.8 % of strain for virgin material. For further deformation (stage-III, upto 5.8% of strain in virgin material) the formation of more martensite exerted internal stress. The dislocation movement was restricted leading to pinning of domain wall movement. This led to slow decrease in coercivity. If the strain was increase further, decohesion of grain took place. At the beginning of such decohesion, stress relaxation occurred which decreased the coercivity further (stage-IV, upto 7.2% strain of virgin sample). As soon as the strain increased, void formation took place that again acted as pinning point and hence, increase in coercivity was observed (stage-V). SEM fractographs were taken and shown in fig-4. Fractograph of virgin sample indicated extended voids with more fibrous structures (fine network of dimples) showing ductile fracture with gradual magnetic hardening (coercivity rise).
Fig-1: Optical micrograph of 304SS samples in the state of (a) Virgin, (b) 12% and (c) 17% Martensite contained.
The cold worked samples exhibited similar behaviour as in virgin one with different extent of % strain in various stages as shown in fig.-3. The stage-I extended to 1.02% and 0.26% strain for initial martensite content of 12% and 17% respectively. The larger volume fraction of these isolated martensites and their pronounced domain wall pinning led to rapid increase in coercivity than the virgin sample. On further straining (stage-II) the growth of martensite took place much faster in higher cold rolled samples and hence showed early reduction in coercivity. These rapidly growing martensites owing to their higher specific volume, induced greater stresses to impede domain movement and was observed with significant rise in coercivity in stage-III. On subsequent increase in tensile loading (stage-IV), the extents of decohesion and its stress relaxation (coercivity decrease) was less due to higher initial martensite content. The greater volume fraction of martensite not only impeded dislocation movement in this stage but also led to restricted domain wall movement with coercivity increase in stage-V. The reduced dislocation movement decreased the size of voids as the initial martensite content increased from 12% to 17% as shown in fractographs (fig-4).

The response of the Barkhausen emissions with the formation of matertensite at low strain was not as sensitive as from hysteresis loop measurement. MBE signal was not observed at all at stage I of the virgin sample where the martensite grains were situated apart. MBE signal was observed only at the end of stage-II where the martente grains are interacting with each other strongly. The presence of higher percentage of martensite exerted internal stress that restricted the dislocation movement. Unlike coercivity, Barkhausen voltage increased in stage-III that apparently showed magnetic softness. This increase of MBE signal was due to more martensite development as well as increase of low amplitude pulses coming from the pinning of domains by dislocations. The stress relaxation in stage-IV allowed the dislocation movement and subsequently reduced the number of low
amplitude pulses and hence decreased rms voltage. The formation of voids in stage-V again increased the low amplitude pulses that led to slow change in MBE voltage.

Fig-3: Variation of Magnetic coercivity with tensile loading
In cold worked sample, the presence of initial percentage of martensite played a major role in Barkhausen emissions. After an initial increase MBE voltage become almost constant within with stage-I and initial part of stage-II of cold worked sample having 12% of martensite. The internal stress generated due to more martensite formation decreased the MBE signal. The increase in MBE voltage before fracture was due to the relaxation of stress as well as the formation of voids. Similar nature was also observed for 17% cold worked sample.

Fig-4: Variation of Magnetic Barkhausen Emission (MBE) rms voltage with tensile loading
Fig-5: Fractographs of (a) Virgin, (b) 12% and (c) 17% Martensite contained 304SS samples
4.0 Conclusion
Non-destructive evaluation techniques like Magnetic hysteresis loop (MHL) and magnetic Barkhausen emission (MBE) measurements could be used to study the tensile behaviour of virgin and cold rolled 304SS samples. The variation in coercivity and Barkhausen voltages obtained from MHL and MBE respectively exhibited different stages of plastic deformation. Barkhausen was able to pick-up the surface sensitive changes to very low deformations that affected relaxation of internal stresses and its associated magnetoelastic as well as magnetostrictive behaviour. However, this technique was unable to make an early detection of martensite generated during initial tensile loading below yield point. Coercivity measurement could detect even low volume fraction of martensite generated in the materials bulk of virgin as well as cold rolled specimens.

References