Application of electrical conductivity as an NDE tool for microstructural evaluation during cold rolling and thermal ageing

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Abstract

The application of electrical conductivity as a non destructive technique for understanding the microstructural evolution during cold rolling and thermal ageing is demonstrated in this study. In general, the electrical conductivity depends on density of dislocations, precipitates and crystal structure of the matrix and is influenced by changes in these microstructural features. Dislocations and precipitates are lattice imperfection in crystalline materials which decreases the electrical conductivity by scattering effect. Similarly, the electrical conductivity is more in a closed packed structure as compared to an open crystal structure.

In the present study, stainless steel specimens of grade SS316 have been cold rolled up to 60% reduction in thickness in steps of 5%. The 40% cold worked (CW) steel specimens are further annealed at different temperatures between 300 °C to 1200 °C. The two sets would enable in understanding the changes in dislocation structure. Electrical conductivity measurements were carried out on these two sets of specimens. The investigation showed decrease in electrical conductivity with increase in the cold work of 316 SS specimen. The XRD peak profiles (311) of the above specimens were analysed to estimate the microstrain. The observed variation of electrical conductivity, microstrain and changes in hardness are explained.

Similarly, changes in electrical conductivity of Ti-alloy heat treated below β -transus temperature (BTT) is investigated. The phase transformation of β Ti-alloy (body centred cubic) to α Ti (hexagonal closed pack structure) on ageing is analyzed from conductivity measurements and the conductivity is shown to be proportional to α phase fraction.

1. Introduction

Material properties of metals and alloys, viz., electrical conductivity, thermal conductivity, magnetic permeability, density and elastic modulus vary due to cold rolling, thermal ageing and other heat treatments. The change in strength of materials is achieved by cold rolling, modifying the grain boundaries, precipitation or by adding fine dispersoids. The variation in material property on strengthening is attributed mostly due to the influence of dislocation density, precipitates, grain size and crystal structure of the matrix. By determining these material properties by NDE techniques, it is possible to characterize the microstructural changes during the strengthening process. There are many NDE techniques like eddy current, potential drop,

ultrasonics, and magnetic Barkhausen emission (MBE) techniques which are dependent on these material properties. The eddy current and potential drop techniques are dependent on electrical conductivity and permeability. However, the eddy current technique is more suitable for non magnetic materials as the effect of both magnetic permeability and conductivity cannot be separated easily[1]. The eddy current technique is a non contact NDE technique, which can be used to estimate the electrical conductivity in addition to its application for defect detection. Non destructive characterization of ageing process, like precipitation hardening during isothermal ageing and fatigue life assessment has been carried outusing changes in electrical conductivity, thermal conductivity and hardness[2,3]. Similarly characterization of strain induced martensite during cold working of SS 304 has been carried out using MBE and SQUIDs[4].

In the present study, the applications of electrical conductivity to understand the hardening that occurs during cold working (CW) of 316 SS to various levels and softening behaviour due to annealing of 40% CWare discussed. In addition to this, the application of electrical conductivity measurements in Ti-alloy to determine the phase fractions would be elucidated.

2. Experimental details

316 SS plateswere cold worked up to 60% reduction in thickness in steps of 5%. These plateswere cut to dimensions 25 x 25 x 12 mm³ for this study. The set of 316 SS specimens cold worked to 40% were annealed from 300 °C to 1200 °C in steps of 100 °C. Additionally, β -titanium alloy with chemical composition of Ti-10V-4.5Fe-1.5Al were quenched at 900 °C for 1 hour and heat treated at different temperatures from 550 °C to 900 °C in steps of 50 °C. The Ti-alloy specimens were of dimensions 20 x 15 x 08 mm³. The electrical conductivity measurements in these two materials were carried out using SIGMATEST 2.069 instrument. The instrument non destructively measures the electrical conductivity based on eddy current principle. The instrument is calibrated with standard specimens of known conductivity. The hardness was measured using 423D digital Vickers hardness tester.

3. Results and discussions

3.1 Cold working and annealing of SS 316

Electrical conductivity and hardness measurements were carried out based on the procedures mentioned earlier. The variation in hardness and electrical conductivity of cold worked 316 SS specimens are shown in Figure 1. Figure 1 shows the increase in hardness and decrease in conductivity as the percentage of cold working is increased from 5% to 60%. The conductivity of virgin 316 SS specimen is 1.30MSi/m.The increase in hardness with CW% shows a typical two slope behaviour with initial increase from 185 to 306 VHN when the CW level reaches 25%. Similarly, the conductivity drops to from 1.30 to 1.25 MSi/m. Further increase in CW level increases the hardness at a lower rate and reaches a maximum of 383 VHN at 65% CW. However conductivity drops drastically as compared to the initial regime to a value of 1.06 MSi/m. A guide line is drawn in Figure 1 to indicate these changes. The decrease in conductivity is based on the Matthiessen's rule[5] which states that resistivity is a summation of intrinsic resistivity, temperature dependence of resistivity and variation due to defects. It is well known that during CW, increase in dislocation density occurs followed by cell structure formation which causes the reduction in conductivity[6]. The observed changes in hardness are in accordance to these changes. However, it is observed that conductivity changes during higher

levels of cold working are not linear. The drastic decrease at 35% CW is due to the cell structure formation which impedes the electron motion causing a reduction in conductivity. Further drop in conductivity is attributed to formation of fresh dislocation on further CW up to 65%. The observed variation of conductivity shows that the variation in the electrical property can be used to understand the cold rolling behaviour.



Figure 1.The variation of electrical conductivity and hardness with respect to percentage of cold work of 316 SS



Figure 2. The variation of electrical conductivity and hardness with respect to annealing temperature of 40% CW 316 SS

The variation of hardness and conductivity of 40% cold worked 316 SS annealed at various temperatures is shown in Figure 2. The change in hardness with ageing temperature shows three different slopes/regimes, indicating recovery, re-crystallization and grain growth. These regimes

are also observed in conductivity measurements. However, the conductivity changes during recovery and grain growth are marginal. The change in conductivity is drastic in the recrystallization regime, similar to the large changes in hardness. The variations in electrical conductivity during cold working and annealing showed an inverse relationship with hardness.



Figure 3. The variation of microstrain and hardness of 40% CW 316 SS with annealing temperature

To understand the softening behaviour of 40% CW SS 316, Fourier analysis of (311) peaks have been carried out based on the approach proposed by Nandi et al.[7]. The details of these observations are presented elsewhere[8]. From this analysis, microstrain has been determined and the variation of hardness and microstrain with increasing annealing temperature is shown in Figure 3. The figure clearly shows that there is a reduction in microstrain as the annealing temperature is increased, which indicates softening of the matrix caused by the annihilation of dislocations. This observation is in agreement with the reduction in hardness.

3.2 Studies in Ti alloy for phase fraction determination

It is well known that conductivity depends on the crystal structure which determines the availability of conduction electrons in any material. To understand the effect of changes in crystal structure on electrical conductivity, the conductivity measurements have been carried out on Ti-alloy. The β -transus temperature (BTT) of this alloy is 780 ± 10 °C.The minimum temperature at which the Ti-alloy shows complete β phase is said to be the BTT.The crystal structure of Ti-alloy in β phase corresponds to body centered cubic (BCC) structure whereas the α phase corresponds to hexagonal closed packed(HCP) structure. When annealed at temperatures below BTT, α phase forms and the variation of conductivity with annealing temperature is shown in Figure 4. The α phase fraction has been determined from JMatPro simulation software and has been quantified with XRD phase analysis[9]. Figure 4 also shows the variation of α phase fraction determined from simulations. The Ti-alloy shows β phase at higher annealing temperature above BTT and when the annealing temperature decreases, the percentage of α phase increases.



Figure 4. The variation of conductivity and alpha phase fraction (%) with annealing temperature of Ti-alloy



Figure 5. The variation of conductivity with alpha phase fraction in Ti-alloy

Figure 5shows the variation of electrical conductivity with percentage of α phase of Ti-alloy. The observed linearity showed an \overline{R}^2 value of 0.992 which shows that conductivity can be used to estimate the phase fraction. It is also observed that, the electrical conductivity is lower for body centered cubic system and higher for closed pack cubic system. In this study the crystal structure dependent electrical property of the Ti-alloy has been used to estimate the various phases present in the alloy.

4. Conclusions

- The electrical conductivity decreases during the cold working of SS 316. The 40% coldworked specimens showed an increase in conductivity with increase in annealingtemperature.
- The hardness and the conductivity variation were found to be inversely related during the strengthening and softening of SS 316. The conductivity is found to be sensitive to strengthening and softening that occurs due to changes in microstructure.
- ➤ The study reveals that the electrical conductivity is influenced by the microstructural changes due to re-crystallization (at 600 °C), much earlier than the hardness (at 700 °C).
- During heat treatment of Ti-alloy at lower temperatures of BTT, the structural change occurs from body centered cubic system to closed pack cubic system. The higher electrical conductivity of alpha (hcp) phase has been utilized nondestructively to estimate the phase fraction in Ti-10V-4.5Fe-1.5Al alloy.
- The percentage of alpha phase fractionin Ti-alloy is linearly correlated with the electrical conductivity.

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