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NONDESTRUCTIVE CHARACTERIZATION OF THERMAL AGEING BEHAVIOUR AT 753 K IN M250 MARAGING STEEL

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ABSTRACT

Maraging steel M250 is widely used in aerospace industry for fabrication of critical rocket motor casings. Specific heat-treatment is given to these parts before they are put into service. Qualification of the heat treatments is presently based on the microscrostructural examination carried out on test coupons, which is indirect tedious, destructive in nature and only limited sampling based. For the complete, faster, accurate and insitu qualification of the actual heat treatment given to the actual component, non-destructive inspection techniques based methodology has been developed. Two NDE techniques, magnetic Barkhausen emission (MBE) and ultrasonic techniques have been used for the characterization of thermal ageing behaviour of the steel at 753 K. The MBE technique has been chosen due to the ferromagnetic nature of the maraging steel. While the MBE technique provides the near surface information, the ultrasonic technique characterizes the bulk of the component. The present study has clearly brought out the complementary nature of these two techniques for the characterization of thermal ageing behaviour in M250 maraging steel. Ultrasonic measurements have been found to be more sensitive to the precipitation of intermetallics, whereas, MBE could clearly identify the onset of austenitic reversion.

Keywords: Maraging, NDT, Ultrasonic, MBE

1. INTRODUCTION

Maraging steels are low-carbon iron nickel martensitic steels developed in the 1960s for applications requiring ultrahigh strength combined with good fracture toughness[1]. These steels contain 18 wt pct Ni, together with substitutional elements such as Co, Mo and small amount of Ti. The most common grades of the 18 Ni grades are M200, M250, M300 and M350, where the numerical designation represents the yield strength (in KSI) of the steel grade. Among these, M250 grade steel is widely used in the manufacture of critical rocket motor casings of the space industry. The high yield strength [1700 MPa] coupled with good fracture toughness [120 MPa$\sqrt{m}$] makes this steel attractive for such demanding applications. These steels are used for the heavy-duty structural applications in quenched and thermally aged microstructural condition. Excellent mechanical properties are obtained by the precipitation of intermetallic phases in the low carbon martensitic matrix by using optimum heat treatment conditions[2]. The strength in aged condition is derived from the fine and coherent intermetallic precipitates, where as low carbon martensitic structure provide the high fracture toughness. Over-aging results in coarsening of the intermetallic precipitates in addition to the reversion of martensite to austenite. This duplex process due to overaging affects both tensile and fracture properties of these steels. Hence, there is always a stringent requirement for the qualification of these heat treatments. The qualification of these critical parts is currently based on the metallurgical examinations carried out on the coupons heat treated along with them and assuming that coupons possess similar microstructure as that of the component. This assumption may not be true always. Hence, it becomes necessary to qualify the component as a whole based on the investigations on the component itself rather than using test coupons. Nondestructive testing techniques such as magnetic barkhausen emission (MBE) and ultrasonic velocity show good promise in this regard.

Ultrasonic velocity measurements have been used to study the precipitation behaviour in different alloy systems by various researchers. In Nimonic alloy PE 16, Jayakumar et al.[3,4] correlated the ultrasonic shear and longitudinal wave velocities with the variations in density and elastic moduli due to occurrence of secondary phases. The velocities increased linearly with increase in the volume fraction of γ' precipitates in this alloy. In 9Cr-1Mo ferritic steel, Anish Kumar et al.[5] have found that the ultrasonic velocity of the normalized and tempered alloy increased with the precipitation of fine Cr_2N and Fe_2Mo precipitates and decreased with their coarsening and dissolution. Ultrasonic parameters have been used extensively for the characterization of precipitation behaviour in aluminum alloys [6,7].

In maraging steels, Vary et al.[8] has correlated ultrasonic parameter with fracture toughness in different microstructural conditions, however to the best of our knowledge, no study has been carried out so far for characterising the ageing behaviour of maraging steel through ultrasonic velocity measurements. As the thermally aged M250 maraging steel possess complex microstructures containing intermetallic precipitates of Ni_3Ti, Fe_2Mo and austenite in the martensitic matrix, an attempt has been made to study the influence of aging treatment and the associated microstructure on the ultrasonic velocity.

Various microstructural features such as dislocations, grain boundaries and second-phase precipitates strongly affect the domain wall movement and inturn the magnetic parameters[9–12]. Quantitative correlations have been obtained between volume fraction of secondary phases with magnetic parameters (H_c)[13-16]. Similarly, *MBE has* been correlated with volume fraction second-phase precipitates [17-20]. D.K. Bhatacharya et al.[21] have studied the thermal aging behaviour in 17-4PH steel. In the quenched condition, this steel has soft lath martensitic structure. Upon aging in the temperature range of 750-900 K, finely dispersed copper rich coherent phase precipitate. With increased aging time, loss of coherency and form austenite reversion take place. Each of these stages could be identified through the MBE measurements. The MBE peak decreased initially due to build up of compressive stresses in matrix due to coherency strain associated with copper precipitation, which hinders domain wall movement. Subsequently, loss of coherency due to growth of copper precipitates leads to increase in the MBE signal due to lower resistance to domain wall movement. At longer durations, due to precipitation of paramagnetic austenite phase, MBE signal decreases again. No study has been carried out so far for studying the precipitation behaviour and austenitic reversion using MBE measurements in maraging steels.

Hence attempts have been made in this study to evaluate the variation in both MBE and ultrasonic velocity parameters with aging time at 753 K for different durations. The study is aimed at comprehensive non-destructive assessment of the microstructural changes for qualifying the heat-treatment before putting the component to service.

2. EXPERIMENTAL

Chemical composition (wt %) of the maraging (M250) steel used in this study is as follows: 17.89 Ni, 8.16 Co, 4.88 Mo, 0.43 Ti, 0.05 Mn, 0.05 Cr, 0.05 Si, 0.05 Cu, 0.096 Al, 0.003 C, and balance Fe. A 15 mm thick plate of maraging steel (M250) was solution annealed at 1093 K for 1 h followed by air cooling. The samples in the form of cuboids (30 mmx25 mmx7 mm), made from the solution treated plates, were encapsulated in quartz tubes under vacuum and aged at 753 K for different durations of 0.25, 1, 3, 10, 20, 30, 40, 70 and 100 h followed by water quenching. For ultrasonic measurements, surface polishing of these samples was carried out to obtain a thickness close 7 mm and plane parallelism to an accuracy of ± 2 μm. The same samples were utilized for MBE and hardness measurements also. The Vicker's hardness measurements were carried out at 10 kg load. An average of five hardness measurements was taken for each specimen.

2.1 Ultrasonic measurements

Figure. 1 shows the schematic of the experimental setup used for ultrasonic measurements. Ultrasonic velocity was measured at room temperature using 15 MHz longitudinal and 5MHz shear wave transducers. 100 MHz broad band pulser-receiver (M/s. Accutron, USA) and 500 MHz digitizing oscilloscope (M/s. Lecroy) were used for carrying out the ultrasonic measurements. Cross correlation technique has been used for precise velocity measurements. For the velocity measurements, the ultrasonic signals were digitized at 500 MHz and the gated backwall echoes from the oscilloscope were transferred to the personal computer with the help of General Purpose Interface Bus (GPIB) interfacing and LabVIEW software. The accuracy in time of flight measurement is better than 1 ns and the maximum scatter in the ultrasonic velocity is ± 2.5 m/s.

2.2 MBE Measurements

A U-shaped electromagnet assembly was used to magnetize the maraging steel specimen fixed between two conical pole pieces of an electromagnet. The maximum field was set to 1500 Oestards for complete saturation. This corresponds to a magnetization field strength (H) of 8,00,000 A m^{-1}. A linear bipolar triangular symmetrical time varying magnetic field of 0.4 Hz is generated by a high current amplifier with associated electronics. The Magnetic field (H) parallel to the specimen axis is measured by a Hall probe positioned tangentially very close to the specimen. The MBE signals are picked up by an encircling sensor coil of 5000 turns. The MBE signal is amplified by a low noise preamplifier and a post amplifier (80dB).The amplified voltage is fed to a root mean square (RMS) converter with an adjustable time constant. All the parameters, after suitable conditioning, are fed to a computer based data acquisition system.

3.RESULTS & DISCUSSION

3.1 Ultrasonic Velocities

Figures 3 a and b show the variations in hardness and ultrasonic velocities (longitudinal and shear respectively) with aging duration. The variations in the ultrasonic velocities exhibited similar trend as that of the hardness. They initially increased with aging time, peaked at intermediate ageing duration followed by continous decrease. The initial increase in hardness and ultrasonic velocities up to 3 h is attributed to the precipitation of primary Ni_3Ti intermetallic precipitates from the martensitic matrix. The increase in the hardness is attributed to the precipitation hardening phenomenon. The increase in the velocities is attributed to the increase in modulus of the matrix due to depletion of solute elements (Ni and Ti) from matrix during precipitation of Ni_3Ti. This can be shown by comparing the Young's modulus of pure iron and M250 steel. Young's modulus of pure Fe is much higher (214 Gpa) and as the alloying elements are added for example in M250, the modulus reduces to 177 Gpa.

Hence increase in the Young's modulus which inturn affect the ultrasonic velocity is expected upon precipitation of different phases due to depletion of the elements. The continuous increase in the hardness and the accompanied increase in velocities at intermediate duration (10-40 h) are attributed to additional precipitation of fine Fe_2Mo precipitates from the solid solution. Hardness and velocities increase further to a maximum and decrease at longer aging durations. The decrease in the velocities and the hardness upon longer durations is attributed to the formation of reverted austenite with lower hardness and velocities. This austenite has been reported to be soft phase having lowest moduli among all the phases present[22].Though the reversion of austenite is reported to start much before 40 h, the decrease in the hardness and the ultrasonic longitudinal wave velocity is observed only after 40 h. This is attributed to the fact that the precipitation of Fe_2Mo which tend to increase in the hardness and velocities also goes in parallel with reversion to austenite. Hence decrease in hardness and ultrasonic longitudinal wave velocity due to formation of austenite can be felt only when this decrease is more than the increase in these parameters due to continued precipitation and for growth of intermetallics to optimum size. Further, it has been found that the longitudinal wave velocity is influenced more by the reversion of austenite, where as the precipitation of Fe_2Mo influences shear wave velocity more than longitudinal wave velocity. This can be seen by comparing the ultrasonic velocities in martensitic and austenitic steels. The difference in the ultrasonic longitudinal wave velocity in these steels is much larger as compared to that in shear wave velocity. For example ultrasonic shear wave velocity and ultrasonic longitudinal wave velocity in austentic stainless steel are 5690 m/s and 3150 m/s respectively. Whereas they are 6000 m/s and 3300 m/s respectively for 9Cr-1 Mo ferritic steel.[23] The larger influence of precipitation of intermetallics on shear wave velocity as compared to longitudinal wave velocity can be seen very clearly by total increase of 150m/s (5%) in shear wave velocity as compared to 125m/s (2.1%) in longitudinal wave velocity from the SA condition. The maximum in hardness coincides with the maximum in ultrasonic longitudinal velocity at 40 h whereas shear velocity exhibited the maximum at 70 h. This maximum in shear wave velocity at 70 h shows that even though sufficient amount of austenite is present, its effect on shear velocity is less and the influence of precipitation is seen up to 70 h indicating that precipitation continues even up to 70 hours of ageing at 753 K.

3.2 Magnetic Barkhausen Emissions

Figure 4 shows the experimental set up used for MBE. The variation in MBE RMS voltage with applied magnetic field for the solution annealed (SA) and aged maraging steel samples exhibited single peak behaviour for all the samples. However, the peak height varies with ageing duration. Hence the MBE peak height has been correlated with the changes in the microstructural feature. Figure 4 shows the variation in peak RMS voltage as a function of aging period. The MBE RMS voltage is found to be highest in the SA condition. It decreases slightly after aging for 0.25 h and remained almost constant up to 10h of aging. Beyond 10 h of aging, the MBE RMS voltage decreased sharply.

Solution annealed specimens comprise of high dislocation density (order of 10^{11}- 10^{12} Cm^{-2}).These dislocations act as barriers for the movement of magnetic domain walls resulting in the MBE RMS voltage of (2.2 V). With aging the SA steel, the dislocation density would reduce, which should have led to the increase in MBE RMS voltage. Contrary to this the MBE RMS voltage was found to be almost constant (about 2.0V) for ageing durations between 0.25 h-10 h. This is attributed to the fact that the annihilation of dislocations which increases the MBE RMS voltage is counteracted by the precipitation of intermetallics occurring simultaneously. The net effect is manifested as constant MBE activity.

On aging beyond 10 h, the MBE RMS voltage drastically drops. This is attributed to the initiation of austenite reversion from the martensite. This fine phase of paramagnetic austenite can only be resolved by TEM. The presence of paramagnetic austenitic phase alters the magnetic behaviour including an increase in the number of microscopic pinning sites, which acts as obstacles for domain wall movement. Moreover the reduction in the volume of magnetic domains participating in the magnetization process at a given instant also results in drastic drop in MBE rms voltage.

The effect of paramagnetic austenite phase on MBE RMS voltage is expected to be very much higher than the presence of coarse precipitates. The effect of coarsening of the precipitates is negligible as compared to the reversion of austenite.
It can also be seen from literature that presence of austenite is reported after 10 h itself for steels of similar composition when aged at a temperature of 753 K. Hence the drop in the MBE rms voltage with the initiation of austenite reversion is in good agreement with that reported in literature.

The above discussion clearly revealed that hardness and ultrasonic velocity are better parameters for studying the intermetallic precipitation behavior, whereas MBE would be better parameter to identify the onset of reversion of martensite to austenite during aging process.

4. CONCLUSION

Ultrasonic and MBE studies were carried out for M250 maraging steel aged at 753 K for various durations. Good correlation was observed between the magnetic and ultrasonic parameters and microstructural changes. The microstructural changes took place in two stages: the intermetallic precipitation and austenitic reversion. The ultrasonic velocity was found to have similar behaviour as that of hardness. They continuously increase to a maximum and decrease afterwards. The increase is attributed to the precipitation of the intermetallics Ni_3Ti and Fe_2Mo whereas the decrease is attributed to reverted austenite. Ultrasonic longitudinal wave velocity was found to be more sensitive as compared to shear wave velocity, to austenitic reversion. Ultrasonic shear wave was found to be more sensitive to precipitation as compared to longitudinal wave velocity and growth of intermetallic, in parallel to austenite reversion. On the other hand, MBE RMS voltage was found to be very sensitive to determine the on set of austenitic reversion due to its paramagnetic nature. The present study has clearly brought out the complementary nature of these two techniques for comprehensive characterization of ageing behaviour in maraging steels.

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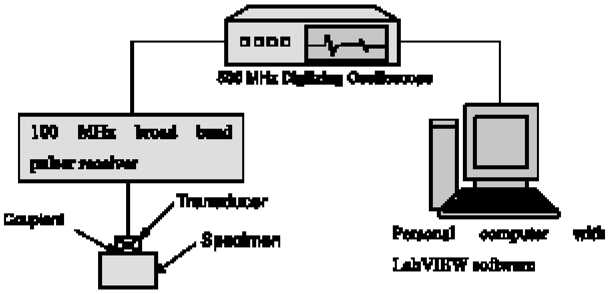

Fig. 1 Schematic of the experimental setup for ultrasonic measurements.

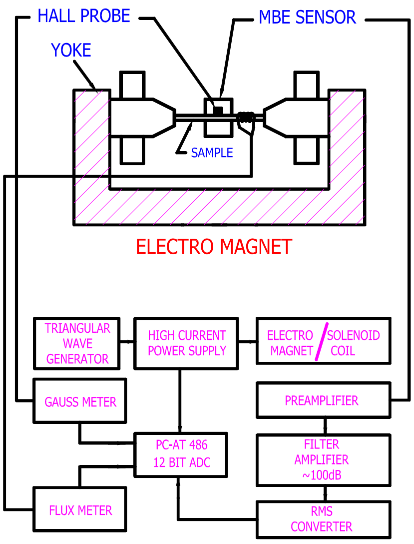

Fig. 2 shows the schematic of the experimental setup used for MBE

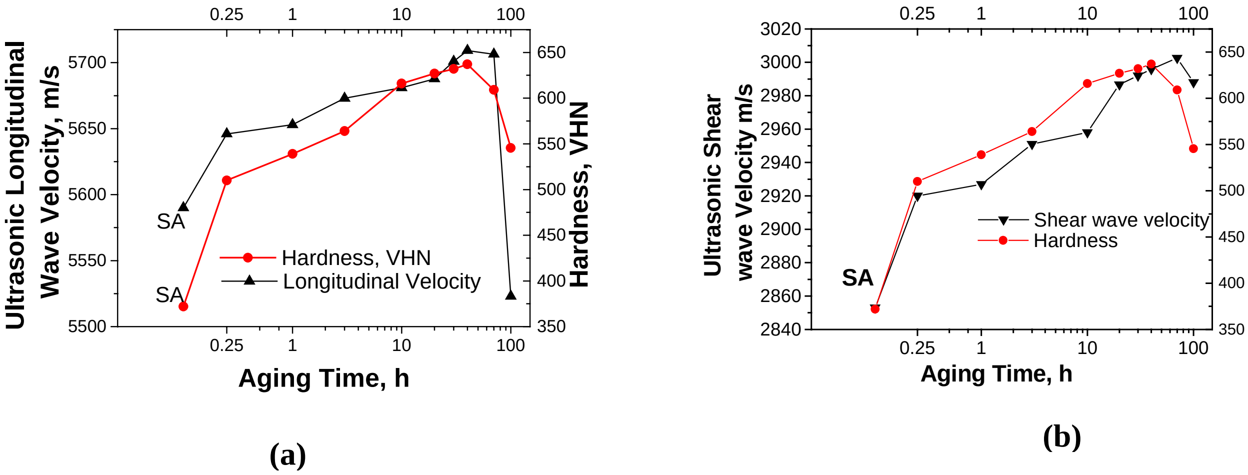

Fig.3 Variation in hardness and ultrasonic (a) longitudinal wave and (b) shear wave velocity with aging time

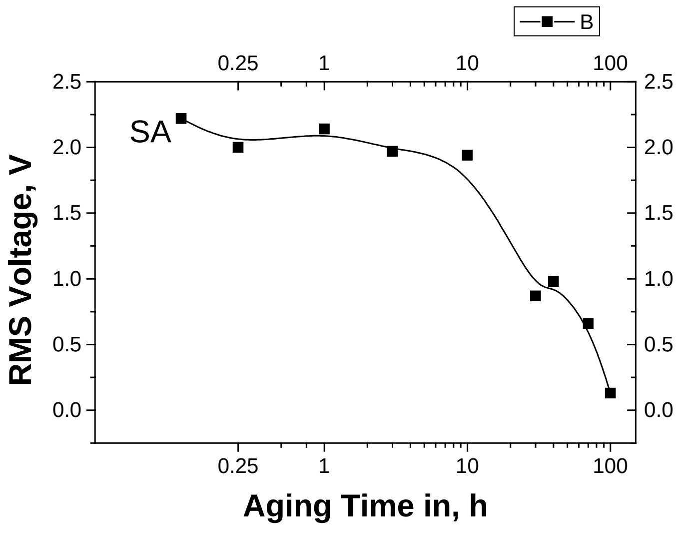

Fig.4. Variation of MBE RMS voltage with aging time