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Aging–induced Microstructural Changes in M250 Maraging Steel using In-situUltrasonic Measurements

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Abstract: Maraging steel (M250) is widely used in many technological sectors like aerospace, military and nuclear power plants. In-situ ultrasonic characterisation is an excellent tool to study the temperature dependent structural/microstructural changes in materials. An indigenous experimental set-up developed in the authors' laboratory is used for in-situ measurements of ultrasonic velocities and attenuation over a wide range of temperatures from room temperature (300 K) to 1200K.The in-situ structural/microstructural changes that occur in maraging steel during thermal treatment are studied employing in-situ ultrasonic velocity and attenuation measurements. The obtained results reveal recovery of martensite, formation of coherent intermetallic Ni_3(Ti,Mo) precipitations, dissolution of Ni_3(Ti,Mo), formation of Fe_2Mopreparation, dissolution ofFe_2Mo, austenite revision and transformation of martensite to austenite.
Keywords: Maraging steel;Ultrasonic velocities; Attenuation;Microstructure.

1. Introduction

Maraging steels are high strength and high fracture toughness steels characterised by intermetallic precipitation in iron–nickel martensite. The excellent mechanical properties i.e., ultrahigh strength combined with good fracture toughness, hardness, ductility and corrosion resistance of maraging steels, makes these steels the most preferred materials for structural applications like aerospace, military and nuclear power plants [1-2]. Furthermore, the dimensional stability and easy heat treatment make these steels an attractive material for use in machinery and tools applications [3].

Similarly, the properties like excellent weldability, goodformability and high resistant to crack propagation, maraging steels are used as efficient materials for various applications like construction of missile and rocket motors, landing gears, wind tunnel models, recoil springs, flexures and AC motorshafts[1-4]. In view of the sensitivity of thermal aging treatments on the mictostructure in maraging steel, particularly to evaluate intermetallic precipitations and austenite revision that influence the strength and fracture toughness, a systematic study on microstructural changes that take place during the aging of maraging steel is carried out extensitively [5]. Most of the studies used for the evaluation of microstructural changes associated with aging of maraging steel are destructive, time consuming and off-line in nature.

Therefore, the temperature dependent in-situmicrostructural characterisation of maraging steel over wide range of temperatures is usedto explorethe microstructural changes in maraging steels during aging. The in-situmeasurement of temperature dependent longitudinal/shear wave velocities, attenuation and derived parameters like elastic constants [6-9] is used to determine grain size [6], volume fraction of different phases, hardness, yield strength, critical temperatures, fracture toughness and creep and fatigue damages [7-9].

In this present study, the structural/microstructural changesthat take place in the maraging steel during aging are studied in-situemploying ultrasonic velocity and attenuation measurements. The observed in-situ variationson the measured parameters are revealed in terms of the microstructural changes take place in maraging steel during aging.

2. Experimental

2.1 Specimen preparation

The chemical composition (wt. %) of the M250 grade maraging steel was 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, Bal. Fe. A bar of maraging steel was cut into a small specimen of size 30 × 25 × 7 mm. The specimen was solution annealed at 1093 K for 1 h followed by air cooling. After air cooling, both surfaces of the specimen were surface ground to have uniform thickness within 3 μm variation. After grinding, the opposite surfaces of specimen were highly polished using standard metallographic procedures, to achieve proper impedance matching and a good contact between the specimen and transducers for propagation of ultrasonic waves into specimen. The plane parallelism between the two opposite faces of sample was checked using a surface plate and a dial gauge.

2.2. Ultrasonic velocity measurements

A high power ultrasonic Pulser Receiver (Olympus NDT, 5900 PR, USA) and a digital storage oscilloscope (Lecroy, Wave Runner 104 MXi, 1GHz, USA) were employed to record digital ultrasonic (rf) signals. Longitudinal and shear waves were generated respectively by X- and Y-cut transducers with a fundamental frequency of 5 MHz.An indigenously designed experimental set-up was employed to measure the ultrasonic velocities and attenuation over a wide arrange of temperatures using through transmission technique, as discussed elsewhere [10]. The measurements were made in the temperature range from 300 to 1200 K at a heating rate of 1 K min^{-1}.The measurement temperature was controlled employing a programmable temperature controller (Eurotherm, 2604, USA), under dynamic mode of operation. The error in measurement of temperature is ± 1 K. At a given time, by measuring transit time t_1 only with buffer rods and then transit time t_2 by introducing the specimen in between buffer rods, the difference in the transit time (Δt) was determined. Thus, the ultrasonic velocities (U_L/U_S) were determined using the relation [8]:

$$U = \frac{d}{\Delta t} \qquad (1)$$

The overall accuracy obtained in the measurement of the velocity is ± 5 m s^{-1}.

2.3. Attenuation measurements

The attenuation coefficient in the specimen was calculated by measuring amplitudes of echoes in the time domain trace using the following relation [10]:

$$\alpha(f) = \frac{1}{d}\left(\ln T + \ln\left(\frac{A_w(f)}{A_s(f)} \right) \right) \qquad (2)$$

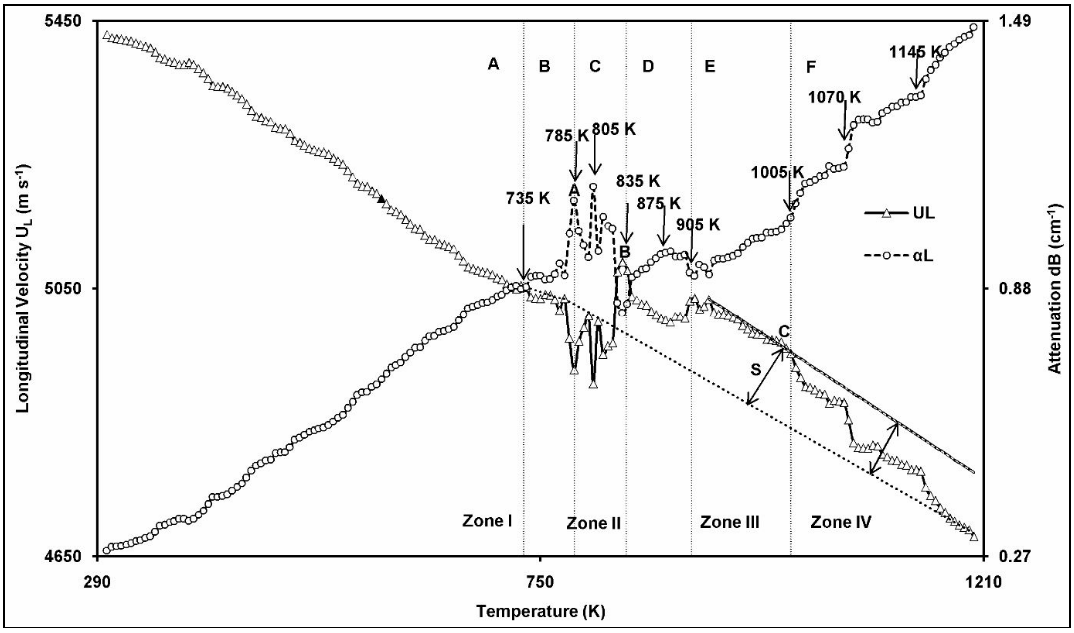
where $A_w(f)$ is the amplitude of the received signal with waveguides only and $A_s(f)$ the amplitude of received signal when the specimenwas inserted between waveguides.

The combined transmission coefficient at the specimen and waveguide interface is

$$T = \frac{4Z_w Z_s}{(Z_w + Z_s)^2} \qquad (3)$$

where$Z_w$and $Z_s$are the acoustic impedances of waveguide and specimen. The percentage error in measurement of attenuation coefficient is ± 2%. In order to provide good contact at interfaces between specimen, waveguide and transducers, a uniform pressure was maintained between transducer and specimen, with suitable arrangements.The couplant correction for measured velocity and attenuation has been carried out using the standard procedure[11].

3. Results and Discussion



Captions for Figure

Fig.1 Temperature-dependence longitudinal velocity and attenuation of M250 maraging steel

The longitudinal ultrasonic velocity and attenuation measured as a function of temperature are represented graphically in Fig. 1. Generally, in any material, a continuous decrease in velocity and an increase in attenuation are observed as a function of temperature [8, 10]. However, in this investigation, the ultrasonic velocity and attenuation as a function of temperature show different behaviours i.e., normal and anomalous behaviours. The microstructural changes in maraging steel during aging cause the observed anomalous behaviour in ultrasonic velocity/attenuation.The change in magnitude of ultrasonic velocity/attenuation is used to explore the different processes, namely recovery of martensite, formation and dissolution of intermetallic precipitates and martensite to austenite transformation, which occur in maraging steel during thermal aging.

The temperature dependent velocity shows a gradual decrease with an increase in temperature from 300 to 735 K. In addition, the rate of decrease in velocity and increase in attenuation increases from 735 to 775 K.Both velocity and attenuation show rapid fluctuations with an increase in temperature from 775 to 835 K. In this region, the magnitude of the fluctuations in velocity and attenuation are appreciable. In addition, longitudinal velocity shows an opposite effect to the normally expected, that is sharp peaks at temperatures 775, 800, 810 and 835 K and a sudden fall at temperatures 770, 785, 805 and 815 K. However, attenuation shows an opposite trend to velocity i,e., a sharp dip at temperatures 775, 800, 810 and 835 K and a sudden raise at temperatures 770, 785, 805 and 815 K. Beyond 835 K, a fall in velocity and rise in attenuation is observed up to 875 K and aging from 905 to 1005. An opposite trend from 905 to 930 K is seen in the variation of velocity and attenuation with temperature. Till 1200 K, the rapid decrease in velocity and increase in attenuation are observed beyond 1005 K. To explain the anomalous behaviour observed in both velocity and attenuation as a function of temperature, the entire temperature region (300–1200 K) is divided into six zones, namely Zone A (300–735 K), Zone B (735–785 K), Zone C (785–835 K), Zone D (835–905 K), Zone E(905–1005 K) and Zone F (1005–1200 K).

The different anomalous behaviours observed in velocity and attenuation with aging time are compared with possible phase transformations that take place in maraging steel materials as shown inTable 1. In Zone A, the observed gradual decrease in velocity and an increase in attenuation are ascribed to the vibrations of thermal phonons and interaction of ultrasonic waveswith thermal phonons [12].Generally, an increase in attenuation and a decrease in velocity are observed with an increase in temperature. In Zone B, the observed ultrasonic anomalous i.e., a small dip and peak in velocity respectively at 750 and 775 K are attributed to recovery of martensitei.e., reduction in point defects induced by quenching process [13]. Further, aging up to 785 K leads to increase in velocity/decrease in attenuations and come to expected state of temperature dependence (indicated in dotted line i.e., decrease in velocity and increase in attenuation with increase in temperature, in Fig. 1). Beyond 785 K, the observed sudden fall in velocity and rise in attenuation in Zone C is attributed tonucleation and formation of $Ni_3(Ti,Mo)$coherent intermetallic precipitates[13-14].The coherent intermetallic precipitation leads to the repartitioning of structural elements in martensite structure and the associated change in material's modulus. The increase in the ultrasonic attenuation is attributed to the growth of precipitates, and accompanying structural and coherency changes via the relaxation process [15]. Therefore, maraging steel hardening effects result in a decrease in velocity and an increase in attenuationin Zone C.

Table 1 Comparison of ultrasonic velocity and attenuation anomalous temperature region and Possible Transformation in maraging steel

Zone	Temperature range	Ultrasonic measurements	Possible transformations
A	300 – 735 K	A gradual decreases in U_L and an increase in α_L	Thermal effects only There is no possible transformation.
B	735-785 K	A Typical decrease in U_L and an increase in α_L.	Recovery of martensite Annealing of Quenched point defects [12]
C	785-835 K	An appreciable fluctuations in U_L and α_L	Formation of the main strengthening precipitations Formation of Ni_3TiMo and Fe_2Mo precipitations [12-18]
D	835-905 K	Gradual decreases in U_L and an increase in $\alpha_L$upto 875 K and followed a reverse trend up to 905 K	Coarsening and subsequent dissolution of Ni_3TiMo Simultaneous formation of Fe_2Mo [4,5]
E	905-1005 K	A small dip in U_L and a peak in α_L from 905 to 930 K and followed by a decrease in U_L and an increase in α_L	Coarsening and subsequent dissolution of Fe_2Mo [4, 5] Initialisation of Austenite revision [12, 14-16, 18]
F	1005 – 1200 K	A sudden decrease in U_L and sudden increase in α_L	Coarsening and revision of austenite [13]

The peak in velocity and dip in attenuation centred at 835 K is observed over a temperature range from 825 to 840 K. This is due to coarsening and subsequent dissolution process of $Ni_3(Ti,Mo)$intermetallic precipitates with increase in temperature. The observed dip in velocity and peak in attenuation from 835 to 905 K (Zone D) is associated with precipitation of Fe_2Mo intermetallic [4, 5]. The coarsening and partial dissolution of globular precipitation of Fe_2Mo occurs during aging of maraging steel in the temperature region connecting to Zone E [5].Interestingly, in Zone F (1005–1200 K), instead of a gradual decrease in velocity and an increase in attenuation, a stepwise decrease in velocity and an increase in attenuation are observed. Beyond aging at 1005 K, the maraging steel gets transformed to austenite from martensite state, and hence, the velocity takes rapid decrease instead of taking a gradual decrease as marked by double dotted line. Therefore, as a temperature increases, a velocity decreases at the same rate as that of observed in Zone A i.e., 300–735 K.

In Zone F, an initial sharp decrease in velocity and an increase in attenuation are due to the starting of austenite revision, and hence, contribute to a sharp decrease in velocity and attenuation. As temperature increases (1005–1200 K), the austenite revisiongets completed [13, 16-19], and hence, the rate of decrease in velocity of steel reaches the expected behaviour (dotted line) as that of Zone A. Interestingly, the rate of reduction in Zone F (i.e., 1005–1200 K) is more than the expected normal reduction (double dotted line) and follows the single dotted line. This relatively fast reduction is attributed to revision of martensite to austenite state[13, 16-17, 20]. Thus, the in-situ high temperature ultrasonic velocity and attenuation measurements during the aging are used to confirm the microstructure changes in M250 maraging steel.

4. Conclusion

The following are the conclusions drawn from the present studies:

- Temperature dependence of both velocity and attenuation show a gradual decrease with an increase in temperature from 300 to 735 K, which is ascribed to raise in temperature.

- The coherent precipitation of $Ni_3(Ti,Mo)$, dissolution of $Ni_3(Ti,Mo)$ and preparation of Fe_2Mo are revealed from the in-situ variations observed in velocity and attenuation during thermal aging.
- A sudden decrease in velocity and an increase in attenuation are observed in the temperature range from 785 to 835 K, which is ascribed to the $Ni_3(Ti,Mo)$ precipitation process that is the formation of intermetallic precipitates.
- The intermetallic precipitation strengthens the hardening of the maraging steel. Temperature dependence of ultrasonic velocity and attenuation measurements show gradual decrease in velocities and an increase in attenuation in the temperature range from 905 to 1010 K. The above results confirm the existence of the transformation of martensite to austenite
- A gradual decrease in velocity and an increase in attenuation in the temperature range from 1005 to 1200 K are attributed to the transformation from martensite to austenite.

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