Investigation of Precipitation Hardening of LM22 by Nondestructive Methods

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Apart from traditional application of nondestructive eddy current technique for determination of discontinuities, the method has been recently used to study aging process of 319aluminum alloys manufactured by casting. In this paper, the application of eddy current and conductivity methods for characterization of aging process has been investigated. A relation between mechanical properties and various parameters such as impedance, phase angle and voltage has been established. This study shows that the best results ($R^2 = 0.784$) can be achieved using 110kHz frequency which leads to reversely relationship between Vickers hardness and normalized impedance, in other words maximum value in hardness-time curve coincide to minimum value in impedance-time curve. Results of hardness, conductivity and transmission electron microscope images are in agreement with this result.

Keywords: Precipitation Hardening; Eddy Current Method; Normalized Impedance; Conductivity; Aluminium 319.

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ABSTRACT BOOK
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Investigation of Precipitation Hardening of LM22 by Nondestructive Methods

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Abstract: Apart from traditional application of nondestructive eddy current technique for determination of discontinuities, the method has been recently used to study aging process of 319aluminum alloys manufactured by casting. In this paper, the application of eddy current and conductivity methods for characterization of aging process has been investigated. A relation between mechanical properties and various parameters such as impedance, phase angle and voltage has been established. This study shows that the best results (R^2 = 0.784) can be achieved using 110kHz frequency which leads to reversely relationship between Vickers hardness and normalized impedance, in other words maximum value in hardness-time curve coincide to minimum value in impedance-time curve. Results of hardness, conductivity and transmission electron microscope images are in agreement with this result.

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Introduction

Precipitation hardening is a kind of heat treatment in which mechanical properties depend on temperature and time of process. Old methods for determining mechanical properties of the part after precipitation hardening usually lead to fail it and it is impossible to check all parts in the production line. Eddy current test as a nondestructive method solve this problem. Effect of composition and structure on mechanical properties and also eddy currents is specified and proven. R.B. james [1] used eddy current to measure resistivity of heat treatable alloys. M. Rosen et al [2] studied the aging process in aluminium alloy 2024 by means of eddy current. N. Bowler et al [3] measured electrical conductivity of metal plates using broadband eddy current and four-point methods. X' Ma et al [4] measured electrical conductivity and porosity of metal foams by eddy current. Kogan L.Kh et al [5] investigated effect of the carbon content on the magnetic and electric properties of thermally treated carbon steels and the possibilities of testing the quality of tempering of articles produced from them via the eddy current method. K.V. Rajkumar et al [6] characterized the aging behaviour in M250 grade maraging steel using eddy current nondestructive methodology. S. Hillmann and et al [7] investigated near-surface residual stress profile by high frequency eddy current conductivity measurement. During the precipitation hardening, secondary phase particles (with different size and distribution) form in the matrix. These particles and amount of them have different effects on electron motion (Subsequently on conductivity) so that a special conductivity value and mechanical properties are obtained at special time (Assuming a constant temperature).

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On the other hand, the eddy currents are sensitive to conductivity of material. So it is possible to establish a relation between parameters of precipitation hardening process and output of eddy current test.

Experimental Procedure

In this study a heat treatable aluminium alloy (ASTM A319) has been used. The chemical composition of this alloy is given in table 1. Molten alloy with temperature of 750℃ was prepared in a graphite crucible and inside an electric furnace. After immerging aluminum Degas tablets, pouring was done in a cylindrical cavity with 15mm in diameter and 120mm in length. In order to reduce porosities in the sample, the mould was preheated to 400℃. Seven cylindrical specimens, finally, prepared for heat treatment. In order to produce a homogeneous solid solution, the samples was soaked at 540℃ for three hours and quenched in water. After the formation of a super saturated solid solution, precipitation hardening was carried out at 180℃ for different times. These times are shown in table 2. From The end of each sample, discs with 15mm in length were cut for hardness testing. The auxiliary test in this study was contact electrical resistance measurements that carried out by Valhalla 4300-B Nano ohmmeter. In order to achieve exact results both cross-sectional area of each sample are coated by gold spraying. This coating was very thin. A coil with internal diameter of 18mm and length of 30mm was used for the eddy current testing. A sinusoidal current with a frequency ranging from 10 to 350 kHz was applied. A schematic picture of the used eddy current system is shown in Fig. 1. The eddy current testing was performed at 25℃. For each sample, certain current and voltage signals were used and impendence and phase angle were calculated. Electromagnetic skin depth equation and regression analysis was used for determination of the optimum test frequency. Finally tension test was done on each sample.

Table1. Chemical composition in weight percentage.

Cu	Si	Mg	Fe	Mn	Zn	Ni	Al
3.5	5	0.5	0.6	0.4	0.15	0.15	rest

Table2. Aging time in different samples.

Sample no.	1	2	3	4	5	6	7
Aging time (min)	After quenching	120	195	255	300	330	375

Results and Discussions

Table 3 shows the results of Vickers hardness test. Doing this test on the discs is necessary to ensure the accuracy of heat treatment and these data confirm this. The results of contact electrical resistance test are shown in table 4. Fig. 2 also shows conductivity and hardness results simultaneously. As can been seen, sample 5 have highest hardness in which θ' precipitates have been created. Since the precipitation hardening process has been done according to Previous works, Different stages of deposition are clear [14,18]. Fig. 3 shows the transmission electron microscopy images at different steps of this process and confirm this. Fig. 3(c) shows that after 5 hours, θ' precipitates are obtained. Conductivity changes during precipitation hardening are fully justified. Precipitates are known as “Scattering Center” of electrons, thus smaller precipitates are able to disperse the electrons to higher. With more coarse precipitates, diffraction capability reduced and matrix tends to pure aluminium and increases gradually. This is consistent with early studies [2].

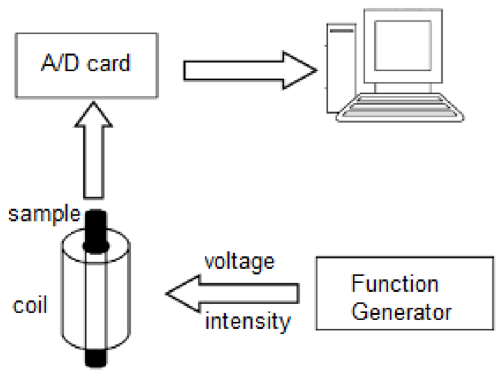

Fig1. Schematic of eddy current testing.

Table 3. The results of Vickers hardness test.

Sample no.	1	2	3	4	5	6	7
Hardness (HV)	68	68	76	78	126	77	63

Table 4. The results of contact electrical resistance test.

Sample no.	1	2	3	4	5	6	7
Resistivity ($n\Omega.m$)	410	440	410	390	370	350	350
Conductivity (%IACS)	42	39	42	44	47	49	49

The difference in conductivity of different samples is the base of eddy current method capability to determine the position in which θ' precipitates has been created. The response of eddy current testing is affected by two major parameters of the sample. These two parameters are microstructure and residual stress [8, 9]. Here discussion is based on microstructure purely.

In eddy current test, determination of optimum frequency is necessary. For this purpose, using regression analysis is easy and many of the limitations of other methods are eliminated [10-13]. In this method relations between different eddy current outputs and hardness in the range of 10 to 350 kHz were studied and best correlation coefficients (R^2 = 0.784) were obtained at 110kHz (Fig.4). Also by using of electromagnetic skin depth concept i.e. equation (1) and considering the sample radius (7.5 mm) as the penetration depth of eddy current, the applied frequency range was determined 128kHz.

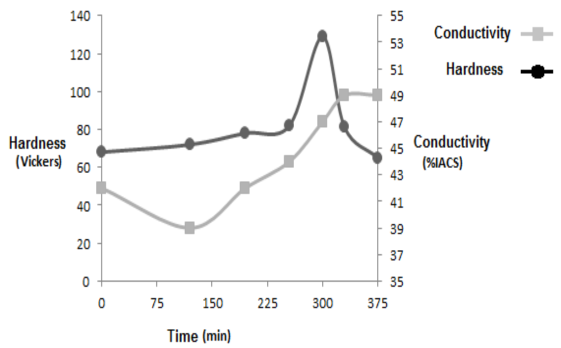

Fig 2. Hardness and conductivity changes during the precipitation hardening process.

$$\delta = \frac{1}{\sqrt{\pi f \mu \sigma}} \qquad (1)$$

Where f is the frequency of function generator in Hz, σ is the electrical conductivity in $\Omega^{-1}.m^{-1}$, μ is the magnetic permeability ($\mu = \mu_{\circ}.\mu_r$; μ_r is the relative magnetic permeability and $\mu_{\circ}$ is the permeability of vacuum i.e. $4\pi \times 10^{-7}$) [15]. It should be noted that this equation is valid for a homogeneous magnetic field parallel to an infinite surface as an approximation.

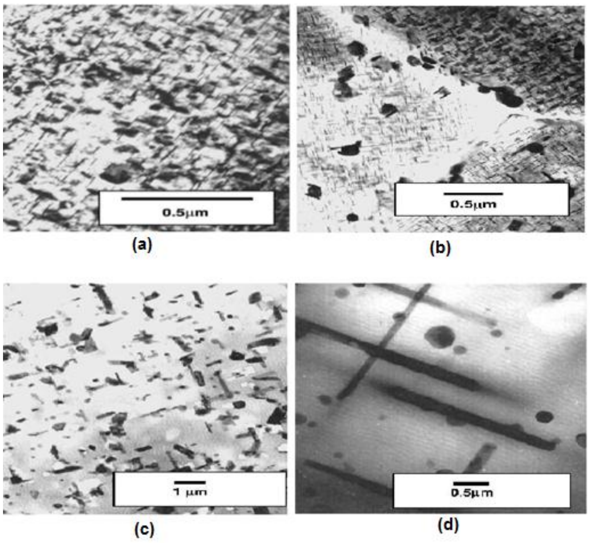

Fig 3. Transmission electron microscopy images show different precipitates during age hardening. (a) Guinier-Preston zones after 120 min, (b) θ'' precipitates after 240 min, (c) θ' precipitates after 300 min and (d) coarse precipitates of θ after 375 min [14].

At frequencies less than 10 kHz and higher than 350 kHz, outputs of eddy current were constant. As a result, considering both mentioned methods, 110 kHz frequency has been used to investigate hardness evaluation by eddy current method.
It is feasible to determine the relation between primary (applied) and induced (secondary) voltage with hardness at 110 kHz frequency but in order to find a better relationship, voltage (V) and intensity (I) of the coil were used to calculate the normalized impedance according to equations (2) and (3).

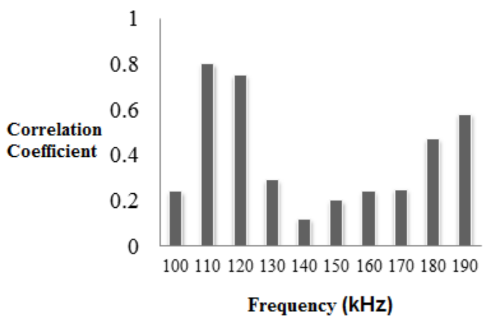

Fig 4. Determination of optimum frequency by regression analysis.

$$Z = \frac{V}{I} \qquad (2)$$

$$\text{normalized impedance} = \frac{Z}{Z_0} \qquad (3)$$

Where Z_0 is the impedance of empty coil. By calculating the normalized impedance for all samples, Fig. 5 is obtained. As can be seen over aged sample has lowest normalized impedance. Matching these data with those obtained from hardness measurements (fig. 6) shows that there is a reversely relationship between Vickers hardness and normalized impedance in other words maximum of hardness coincide to minimum of normalized impedance. This result leads to experimental equation (4).

$$\frac{Z}{Z_0} \propto \frac{1}{HV} \qquad (4)$$

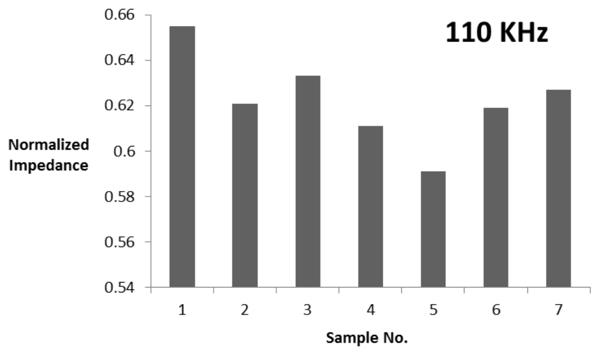

Fig 5. Normalized impedance of samples in which (1) as-quenched, (5) maximum of hardness and (7) over aged.

Where HV is hardness in Vickers. In fact, in the sample have highest hardness (5), good correlation between the precipitate and matrix gradually disappear and atomic disordering increase, this is in turn associated with increasing the hardness and decreasing normalized impedance (or electrical resistance).

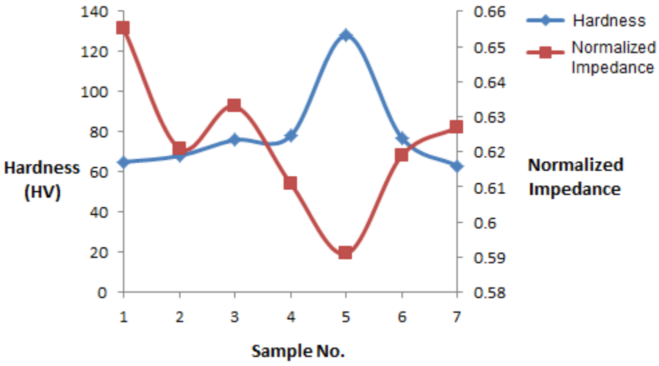

Fig 6. Comparison of hardness and normalized impedance for the samples.

Due to existence of coil in circuit of eddy current, creation of phase shift between voltage and current is inevitable. Using equation (5) and (6), it is feasible to calculate phase shift.

$$Z = \sqrt{R^2 + X_L^2} \qquad (5)$$

$$\theta = Arc\ tg \frac{X_L}{R} \qquad (6)$$

Where X_L is induction resistance, R is resistance and θ is phase shift or phase angle [16]. With achieving phase angle, conductivity can measure by equation (7). In addition, with comparing several samples that aged for different time can recognize the hardest sample.

$$\sigma = \frac{1}{\omega\mu\,(rX(\theta))^2} \qquad (7)$$

Where σ is electrical conductivity (%IACS), ω is period, r is depth of penetration of eddy current and $X(\theta)$ is a function of phase angle[2]. $X(\theta)$ is proportional to the sample geometry, coil type and composition of the sample. Two important relationship that have mentioned for $X(\theta)$ presented in equations (8) and (9).

$$X(\theta) = -0.0018 + 0.0053 \times \theta - 3.6852 \times 10^{-5}\theta^2 - 2.6 \times 10^{-9}\theta^4 \qquad (8) \quad [17]$$

$$X(\theta) = 1.41 \times 10^{-4} + 0.0081 \times \theta + 7.76 \times 10^{-6}\theta^2 - 1.80 \times 10^{-6}\theta^3 + 2.23 \times 10^{-8}\theta^4 \qquad (9) \quad [4]$$

The calculated conductivity with this method has minor differences with what was obtained through contact electrical resistance (about $10^{0.3}$ to$10^{0.7}$). Due to the different conditions which consider in these equations, this is not unexpected. By placing obtained data from experiments, in equations (7), (8) and (9), it is concluded that qualitative and experimental equation (Inverse relationship between hardness and impedance) is in agreement with theoretic equations.

Conclusion

1. Eddy current testing of aluminum alloy, relying on its conductivity, is possible and test's outputs changes are due to different microstructures in various stages of precipitation hardening. samples with maximum hardness have minimum normalized impedance.
2. In order to achieve optimum frequency, two methods have been used (electromagnetic skin depth equation and applied regression analysis) and both methods obtained 110 kHz. for frequencies less than 10 kHz and upper than 350 kHz correlation coefficient is not defined.
3. Calculating of phase angle between current and voltage leads to determination of electrical conductivity of each sample.

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