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Influence of ageing process on sound velocity in C17200 copper–beryllium alloy

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ABSTRACT

This paper has been aimed to explore the influence of ageing process on sound velocity in C17200 copper–beryllium alloy. It was shown that the variation of sound velocity with ageing time follows a two-stage regime, the first stage with a sharp decreasing trend and the second stage developing with an almost constant velocity. Electrical conductivity measurement was applied to provide quantitative evidences to determine the governing mechanism in each stage. It was revealed that the origin of the first stage is the segregation of solute atoms from the matrix, regardless of the ageing temperature. The origin of the second stage, however, was dependent on the ageing temperature. For ageing temperature of 315 °C, increased contribution of the coherent precipitates to the overall elastic modulus of the material was known as the governing factor, and for ageing temperature of 380 °C significant formation of γ equilibrium precipitates was introduced as the responsible factor. Also, it was observed that the correlation between sound velocity and strength of the alloy changes with increase of ageing temperature.

1. Introduction

In recent years, sound velocity measurement has found wide spread use for characterization of materials properties. Since microstructural features (such as precipitates, grain size, dislocations and discontinuities) affect the sound velocity differently, it can be used to provide comprehensive information about the microstructure [1–3], mechanical properties [4,5] and processing histories of the materials [6–9]. Accordingly, finding a deep knowledge of the mechanisms governing the ultrasonic phenomena in different materials and alloys is an essential step to exploit the full potential of this nondestructive technique in materials science and engineering applications.

Despite extensive studies on the variation of sound velocity with the occurrence of precipitation in different materials there is still no unique mechanism which can explain all observations. Murthy et al. [4] observed that in Ni-based superalloy Nimonic 263, sound velocity follows strength and hardness of the alloy with increase in the volume fraction of the coherent γ′ precipitates during ageing. The observed trend was attributed to the change of matrix composition during the first stage of precipitation (i.e. incubation) which influences the elastic modulus of the material. Similar observations have been reported in literature for VT14 titanium alloy [10] and Ni-based superalloy IN 625 [11]. Nishara Begum et al. [12] performed on-line ultrasonic velocity measurements to characterize the microstructural evolutions during thermal ageing of β-quenched Zircaloy-2. They found an inverse relationship between hardness and sound velocity. This was related to the formation of small fraction of hard intermetalics during ageing which partially depletes the matrix from the alloying elements such as Sn, Fe, Ni and Cr, resulting in decrease of the matrix modulus.

Previous studies dealing with the effect of ageing process on sound velocity seem to have one point in common. They all discuss on the effect of matrix composition on sound velocity qualitatively and they represent no quantitative evidence. It is the objective of this paper to explore the influence of ageing process on sound velocity using a quantitative approach. For this purpose, electrical conductivity measurement has been used in correlation with velocity measurement and mechanical testing to determine the compositional changes of the C17200 copper–beryllium alloy during ageing.

C17200 is an age-hardenable alloy and can be heat treated to produce a wide variety of microstructures. It is generally applied for the manufacture of springs, gears, diaphragms and valves due to possessing interesting properties such as corrosion and fatigue resistance, high strength and nonsparking characteristic. In most industrial applications, age hardening of this alloy is done at temperatures between 300 °C and 400 °C for 1/4 h and 4 h at temperature, depending on the properties desired. If the ageing temperature is below 300 °C the precipitation is relatively slow and long periods of time are required to reach maximum properties. If

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Table 1
Chemical composition of the C17200 alloy used in this work.

Element	Be	Pb	Cu
wt%	1.9	0.01	Bal.

the temperature is above 350 °C the precipitation is accelerated, and the hardening effect is produced in a relatively short time. The maximum mechanical properties are obtained by ageing the alloy 1–3 h at 315–345 °C, depending on the amount of cold work [13].

Authors believe that the obtained results can be used to improve the present knowledge of the existing correlations between microstructural features evolved during heat treatment and sound velocity in other materials.

2. Experimental

Table 1 shows chemical composition of the alloy used in this work. Raw materials were provided as sheets of 0.1 mm thickness. Tensile samples were prepared from the as-received materials according to the standard technique (ASTM-A380). Then samples were solution annealed at 780 °C for 10 min followed by water quenching to room temperature. After that, annealed samples were aged for 30–240 min at temperatures of 315 °C and 380 °C. Three tensile samples were tested for each ageing condition using a Gotech-TCS2000 machine and a strain rate of about 0.01 s^{-1}. A CX51 Olympus optical microscope (OM) was used to determine grain size and to study microstructure of the samples. Specimens for OM study were first mechanically polished and then etched using an aqueous solution consisted of 2 g $K_2Cr_2O_7$, 8 ml H_2SO_4 and 4 ml HCl in 100 ml water.

A 20 MHz TR normal probe from Panametrics Co. and a DP25PLUS unit was used to produce sound wave. This unit is an ultrasonic thickness gage which operates on the "pulse-echo" principle. This principle works by precisely timing the reflection of high frequency sound waves from the probe to the far wall of a test sample. The sound waves generated by the probe are coupled into the test sample and reflected back from the opposite side. The same probe then receives the reflected sound waves and converts them into electrical pulses. The gage amplifies the received signal, digitizes a selected portion of the wave train, and then very precisely measures a time interval corresponding to one round trip of the sound waves in the test sample. The measurement of pulse transit time is made between an interface echo marking the time the sound wave enters the test sample and the first back wall echo.

In this work the time of flight was measured with an accuracy of about ±1 ns to determine the sound velocity, leading to the maximum scatter of ±1 ms^{-1} for velocity measurements. Before measuring the sound velocity, exact thickness of the sample in the position of the probe was determined using a micrometer. Then the couplant, ZG-F gel from Krautkramer Co., was applied on the surface of the sample. The measured thickness was given to the gage as the input and the sound velocity was calculated by the gage as the output, using Eq. (1):

$$V = \frac{S}{t} \quad (1)$$

where V is the sound velocity, S is the sound path (two times thickness) and t is the flight time of the wave. On each sample, sound velocity was measured for three times and the average value was reported.

Electrical conductivity of the samples was measured using a Fischer Sigmascope SMP 10 set and an ES40 probe according to the standard ASTM E1004-02 with an accuracy of about ±1.0% IACS.

3. Results and discussion

Fig. 1 shows an OM micrograph of the annealed sample. Grain size of the sample is about 43 μm and found to be constant in all samples; therefore, grain size affect on sound velocity can be ignored [14].

Fig. 2a shows the variation of sample strength with ageing time. It can be seen that strength of the samples aged at 315 °C continuously increases with ageing. For the case of samples aged at 380 °C, initially a sudden increase of strength is observed for the first 30 min of ageing. This increase is followed by a steady decreasing trend. For the ageing times shorter than 120 min samples aged at 380 °C have higher strength than the samples aged at 315 °C and after that the situation is reversed. The precipitation sequence in the Cu–Be system has been shown to be as [13,15]:

Supersaturated solid solution→GP zones→γ''→γ'(BCT)→γ(BCC)

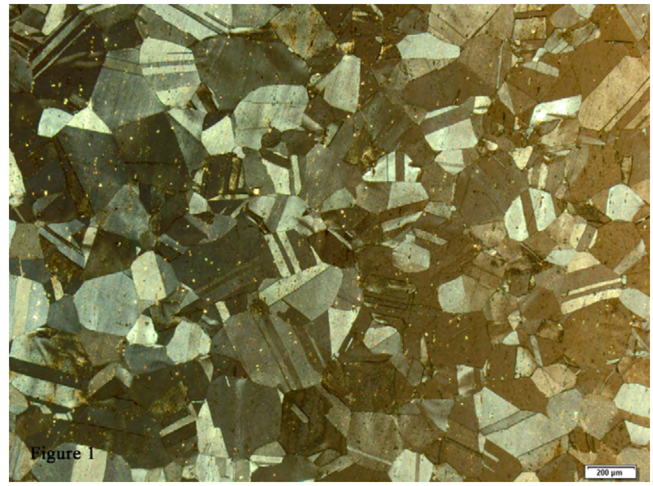

Fig. 1. OM micrograph of the annealed sample.

GP zones are the first precipitates which form coherently on the {1 0 0} matrix planes. With continued ageing, GP zones grow and transform to somewhat thicker ordered plates of γ''. Growth of the coherent precipitates increases the coherency strain fields around the particles and also the lattice distortions. Increase of ageing time and temperature can produce semi-coherent γ' precipitates and noncoherent (equilibrium) γ precipitates. With the increase of coherency, the extension of distortion created by the precipitate within the lattice increases. It is this distortion that retards the movement of dislocations and is responsible for the strengthening of the alloy with ageing. In Fig. 2a, hence, one can relate the observed increase in strength to the formation and growth of the coherent precipitates and the decrease in strength to the loss of coherency and precipitation of the equilibrium precipitates.

As shown in Fig. 2b, variation of sound velocity of the samples with ageing time follows a two-stage regime, regardless of the ageing temperature. At the first stage velocity decreases sharply with increase in ageing time up to 180 min and after that the second stage develops with an almost constant velocity. Sound velocity depends on the elastic modulus of the material through which it is propagating. Formation of different phases during ageing partially depletes the matrix from the alloying elements which, in turn, decreases the elastic modulus of the matrix. As long as the volume fraction of the precipitated intermetalics is small, their contribution to the overall modulus is not considerable and, hence, the net change in the modulus of the material with ageing is due to the change of matrix composition. In Fig. 2b, therefore, the decrease of velocity in the first stage can be attributed to the solute atoms segregation from the matrix. Accordingly, the stop in the fall of velocity with the beginning of second stage is related to the stop in change of matrix composition after 180 min of ageing.

Fig. 2c shows the correlation between sound velocity and strength at the first stage. It can be seen that a linear relationship exists in the first stage for both ageing temperature. Also, it is readily found that the existing relationship changes with ageing temperature, i.e., sound velocity decreases with increase of strength for ageing temperature of 315 °C and decreases directly with strength for the samples aged at 380 °C. The observed change in the relationship is related to this fact that mechanical properties of the alloy (such as hardness, yield strength and ultimate strength) significantly depends on the degree of the precipitates coherency and their fraction which are affected by the heat treatment temperature. This also explains the discrepancy in the reported correlations between sound velocity and mechanical properties of materials by different investigators [4,5,12]. Therefore, care should

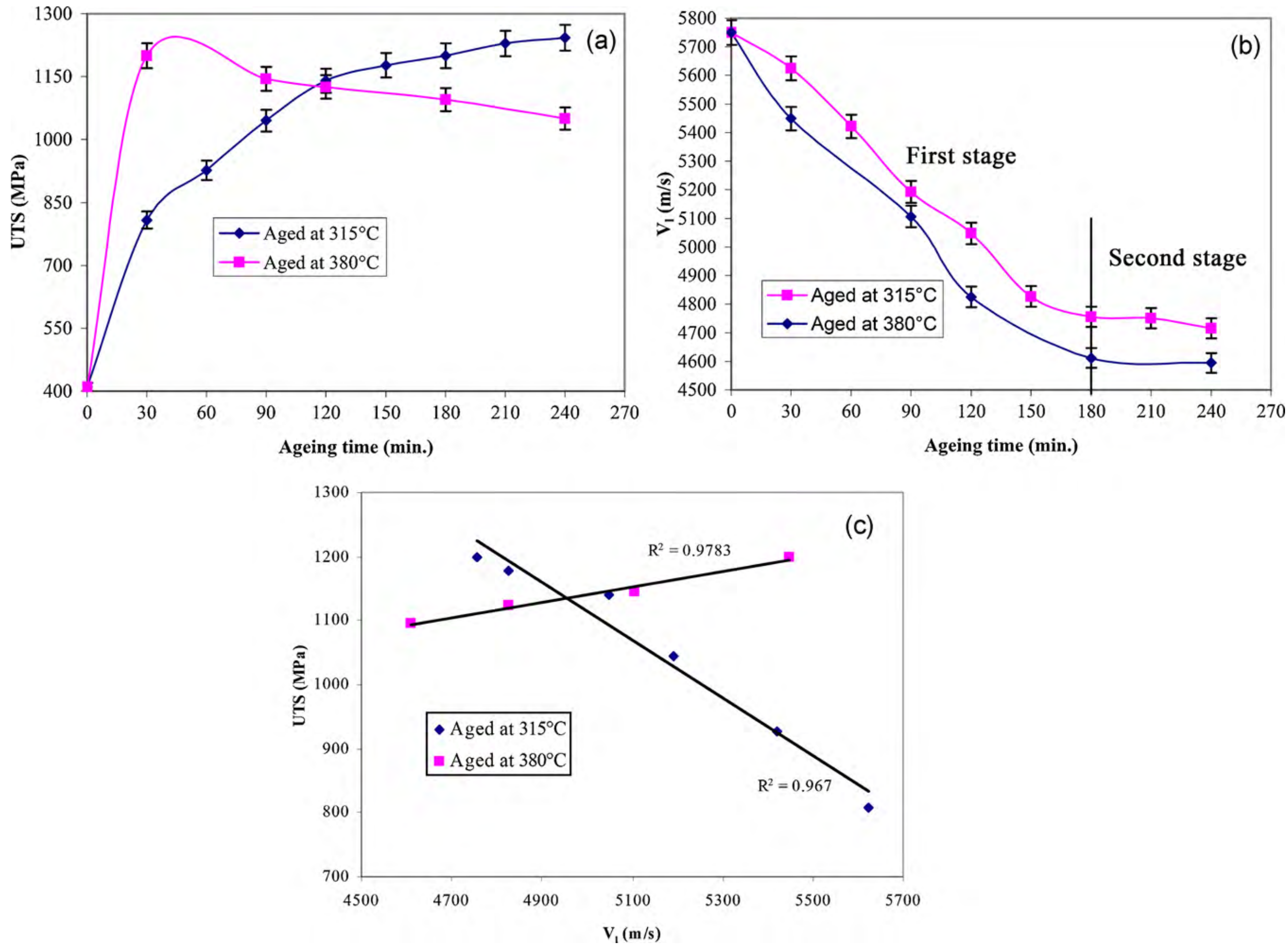

Fig. 2. Variation of (a) strength and (b) sound velocity of the samples with ageing time; (c) correlation between sound velocity and strength of the samples aged at 315 °C and 380 °C.

be taken in determining the variation in mechanical properties of the materials with ageing conditions using velocity measurements.

Fig. 3 shows the variation of electrical conductivity of the samples with ageing time. Electrical conductivity is highly sensitive to the chemical composition and can be used to monitor the compositional evolutions of the matrix during ageing, quantitatively. In fact, the foreign solutes atoms act as scattering centres of electrons and decrease the electrical conductivity of the matrix. As shown in Fig. 3, conductivity of the samples rises increasingly up to 8.1 and

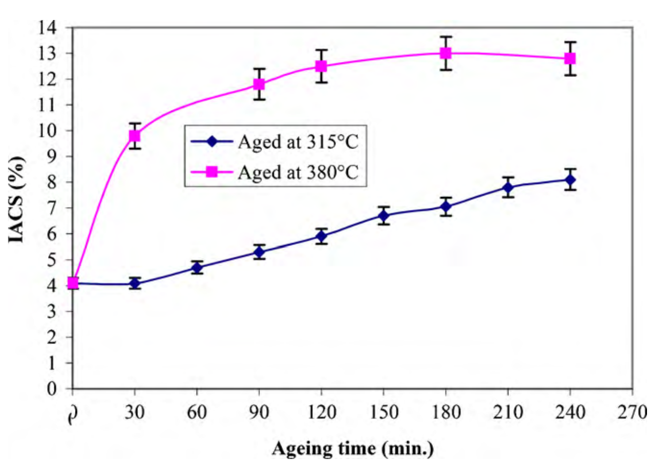

Fig. 3. Variation of electrical conductivity of the samples with ageing time.

13% IACS after 180 min of ageing (end of the first stage) at 315 °C and 380 °C, respectively. The increase of electrical conductivity for both ageing temperatures confirms that purification of the matrix by means of segregation of the solute atoms and formation of precipitates is the origin of the velocity decrease at the first stage (Fig. 2b). In addition, decrease of velocity with increase of ageing temperature (Fig. 2b) can be related to the enhanced removal of solute atoms from the matrix which is manifested by the higher values of conductivity at elevated ageing temperatures.

The striking feature of Fig. 3 is the difference in the trend of electrical conductivity variation at the second stage in regard to the ageing temperature. While conductivity of the samples aged at 315 °C continues to increase, in the case of samples aged at 380 °C conductivity reaches saturation after 180 min of ageing. This difference suggests that the origin of the second stage in Fig. 2b varies with ageing temperature. Continuous increase of electrical conductivity of the samples aged at 315 °C indicates that matrix composition certainly changes with ageing and it cannot be the governing factor in the second stage. Volume fraction of the coherent precipitates and also their elastic modulus increases with continued ageing at 315 °C. Therefore, the influence of coherent precipitates on the material elastic modulus seems to be significant enough at the second stage to stop the velocity to fall more. On the other hand, ageing of the samples at 380 °C provides adequate driving force for the coarsening of γ' phase and its conversion to γ phase. Since these phenomena do not change the matrix composition (as confirmed by the electrical conductivity measurements), the occurrence of the second stage at 380 °C can be mostly

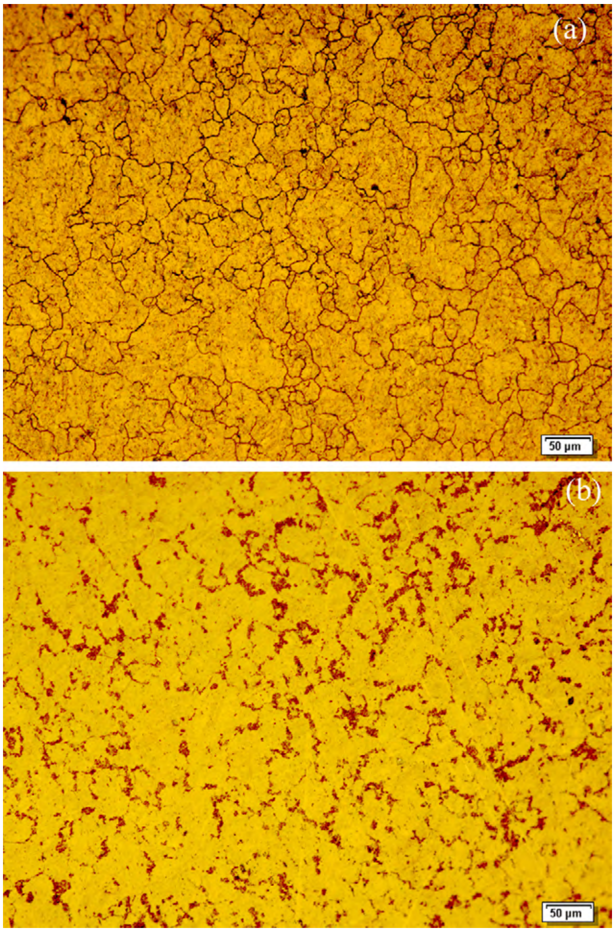

Fig. 4. OM micrograph of the samples aged at (a) 315 °C and (b) 380 °C for 240 min.

attributed to the considerable formation of γ precipitates. Fig. 4a and b shows microstructure of the samples aged at 315 °C and 380 °C for 240 min, respectively. Profound formation of γ precipitates (dark nodules) on grain boundaries is discernable in Fig. 4b.

4. Conclusion

In summary, it was shown that the variation of sound velocity with ageing time in C17200 copper–beryllium alloy follows a two-stage regime, the first stage with a sharp decreasing trend and the second stage developing with an almost constant velocity. It was revealed that the origin of the first stage is the segregation of solute atoms from the matrix which decreases the elastic modulus of the matrix, regardless of the ageing temperature. The origin of the second stage, however, was dependent on the ageing temperature. For ageing temperature of 315 °C, increased contribution of the coherent precipitates to the overall elastic modulus of the material was known as the governing factor. For ageing temperature of 380 °C significant formation of γ equilibrium precipitates was introduced as the responsible factor. Since, formation of this phase does not change the matrix composition, i.e., its formation stop the elastic modulus of the matrix to fall more. Also, it was observed that the correlation between sound velocity and strength of the alloy changes with increase of ageing temperature.

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