Study of ageing-induced α'-martensite formation in cold-worked AISI type 304 stainless steel using an acoustic emission technique

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α'-martensite formation during cooling of cold-worked and aged AISI type 304 stainless steel has been studied by an acoustic emission technique. The ageing was carried out at 673 K for 1 h. A substantial amount of acoustic emission generated during cooling of cold-worked and aged AISI 304 stainless steel specimens compared to negligible acoustic emission observed during cooling (after ageing) of annealed AISI 304, annealed AISI 316 and cold-worked AISI 316 stainless steel specimens, was attributed to the α'-martensite formation from cold-worked 304 stainless steel specimens. The extent of martensite formation was relatively higher for 10% and 50% cold-worked specimens and lower for 20%–40% cold-worked specimens. The temperature range of martensite formation, as detected by the acoustic emission technique lies between 603 and 466 K. The formation of α'-martensite has been established to occur by a shear process.

1. Introduction

Enhancement of the tensile strength of AISI type 304 stainless steel could be achieved by inducing α'-martensite into the metastable austenite by suitable thermo-mechanical treatments. Mangonon and Thomas [1] have shown that ageing of predeformed 304 stainless steel at 673 K increases the amount of α'-martensite and thus improves the tensile properties. They have reported that the yield strength is linearly proportional to the volume fraction of α'-martensite formed, irrespective of its origin, i.e. either mechanical or thermo-mechanical treatments. On-line monitoring of the magnetic phase content during ageing using magnetic methods did not reveal any increase in α'-martensite [2]. Therefore, it was inferred that the enhancement in α' had occurred during cooling from the ageing temperature. Formation of α' during cooling has been attributed to the increase in the M_s temperature of the matrix (with changed composition) to above room temperature after fine precipitation of carbides during ageing [2].

Butler and Burke [3] observed the formation of α'-martensite during cooling in annealed AISI type 304 stainless steel after ageing at temperatures between 873 and 1023 K. The formation of α'-martensite was confirmed by metallographic studies and shown to be the consequence of local increase in M_s temperature due to depletion of chromium and carbon following precipitation of carbides during ageing [3]. They estimated a temperature of 480 K for the initiation of α'-martensite nucleation in annealed AISI 304 stainless steel during cooling after ageing. But the temperature range and lowest temperature up to which α'-martensite formation occurs were not established in their study. Different mechanisms for α' formation have been proposed. Chukhleb and Martynov [4] proposed a nucleation and growth mechanism. Mangonon and Thomas [1] have shown from TEM studies, that new α' crystals are formed by a shear process like martensite formation in Fe–C steels. Butler and Burke [3] observed that α' nucleation during subzero cooling of aged 304 stainless steel was a "haphazard" affair.

Earlier investigations by the present authors [5] showed that the α'-martensite content of cold-worked AISI type 304 stainless steel increased after ageing for 1 h at 673 K. The feasibility of the application of an acoustic emission technique (AET) for monitoring α' formation during cooling of cold-worked 304 stainless steel, was shown.

Prior cold work of the austenite affects α' formation during cooling after ageing. It is reported that small amounts of prior cold work stimulate while extensive cold work suppresses α' formation during subsequent transformation by cooling [6, 7]. Isothermal ageing of partially transformed or cold-worked austenite affects further α' formation by stabilizing the austenite [8]. However, to the best of our knowledge, the effect of prior cold work on the extent of α' formation during cooling of AISI 304 stainless steel after ageing at 673 K has not been studied. No effect of prior cold work on the temperature range of α' formation during cooling is known. Therefore, in this study, an attempt was made to use AET, which has the unique potential

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for on-line monitoring of martensitic transformation [5, 9, 10], to provide better insight into the mechanistic aspects of α′-martensite formation in different cold-worked AISI type 304 stainless steels during cooling after ageing at 673 K.

2. Experimental procedure

The typical chemical composition (wt%) of the AISI 304 stainless steel used in the present investigation is 0.08C, 18.3Cr, 9.7Ni, 1.9Mn, 1.1Si, 0.002S, 0.03P and balance Fe. Solution-annealed (1323 K/1 h) 0.5 mm thick sheet of this steel was cold rolled in the range 0%–50% reduction in thickness at ambient temperature. Coupons, 30 mm × 10 mm in size, were cut from the annealed and differently cold-worked sheets and used for this study. In our earlier study [5], the validation of the experimental apparatus for reliable detection of α′-martensite was already established. In that study, it was also established that the recorded AE signals, if any, are associated with formation of α′-martensite and not due to noise from the experimental equipment by carrying out identical studies on an AISI type 316 stainless steel, which is known to be a stable austenitic stainless steel, and in which transformation to α′-martensite does not take place by either of the mechanical or thermo-mechanical treatments considered in this study. In the present study, the same experimental apparatus as reported in the earlier study [5] was used. The chemical composition (wt%) of the AISI 316 stainless steel used is 0.06C, 17.2Cr, 11.6Ni, 2.2Mo, 0.6Si, 1.8Mn, 0.01S, 0.005P and balance Fe. Ageing of all the specimens was carried out in a horizontal furnace at a temperature of 673 ± 2 K for 1 h. After ageing, the furnace doors were opened and the specimens were allowed to cool inside the furnace, thus maintaining more or less the same cooling rate for all the specimens. The AE signals generated during cooling of the specimens from the ageing temperature were recorded and analysed using an AET-5000 acoustic emission system. AE signals were also recorded during cooling of the furnace without any specimen inside to determine the background noise. A piezoelectric transducer having resonant frequency at 175 kHz, a preamplifier (60 dB gain) and a compatible filter (125–250 kHz) were used to capture the AE signals. Comparison of preliminary results obtained by using 175 and 375 kHz transducers indicated that the signals generated by the 175 kHz transducer were stronger and therefore the 175 kHz resonant transducer was selected for recording the AE signals for this study.

A total system gain of 99 dB was maintained throughout the experiment. The sensor was fixed at one end of a 1.6 mm diameter stainless steel waveguide, the other end of which was spot welded to the specimen. The spot-welded regions were cleaned for all the specimens with emery papers to remove any oxide scale formed during welding. During spot welding, the specimens were covered with wet tissue paper, thus ensuring minimum heating of the specimens. It should be mentioned that magnetic measurements (to determine the starting amount of α′-martensite) were made before and after spot welding in all specimens in terms of equivalent δ-ferrite content. The magnetic measurements were made using a Ferritoscope FE-8 and there was no detectable change in the martensite content due to spot welding. The amount of α′-martensite formed before and after ageing was also estimated in all the specimens. Measurements on three specimens for each cold-worked condition were made to ensure the repeatability. In order to minimize any spatial scatter, measurements were made before and after ageing at the same location, i.e. in the centre of each specimen. The sensitivity of measurement of the Ferritoscope FE-8 used was as follows: 0.1% in the smallest scale (0%–3% equivalent δ-ferrite), 0.2% in the medium scale (0%–10% equivalent δ-ferrite) and 0.5% in the highest scale (0%–30% equivalent δ-ferrite). In all of the measurements of equivalent δ-ferrite content of the cold-worked 304 stainless steel specimens, the scatter observed was 0.2% maximum.

3. Results and discussion

Fig. 1a–d show the variation in root mean square (r.m.s.) voltage of the AE signal with time during cooling (after ageing for 1 h at 673 K) for different specimens. The curves showing the variation in temperature with time are also superimposed on to the same plots. It can be seen from Fig. 1a–d that the AE generated during cooling of the furnace without any specimen (Fig. 1a) and during cooling of annealed AISI 316 stainless steel (Fig. 1b), 50% cold-worked AISI 316 stainless steel (Fig. 1c) and annealed AISI 304 stainless steel (Fig. 1d), is negligible. Estimation of magnetic phase content before and after ageing by equivalent δ-ferrite measurements showed the absence of any detectable α′-martensite in annealed AISI 316, 50% cold-worked AISI 316 and annealed AISI 304 stainless steel specimens.

Fig. 2a–e show the variation in r.m.s. voltage of the AE signal with time during cooling (after ageing for 1 h at 673 K) for different cold-worked specimens of AISI 304 stainless steel. The curves showing the variation in temperature with time are also superimposed on to the same plots. It can be seen from Fig. 2a–e that a substantial amount of AE is generated in the cold-worked 304 stainless steel specimens in the temperature interval 603–466 K during cooling. Below 466 K, a reduction in the intensity (number of peaks in the r.m.s. voltage) of AE generation was observed in all the cold-worked specimens and hence data up to 466 K temperature were used. It is also seen that the intensity of AE is relatively higher for 10% and 50% cold-worked specimens as compared to that for 20%, 30% and 40% cold-worked specimens. The intensity of the acoustic activity is greater in the 50% cold-worked specimen (Fig. 2e) compared to that in the 10% cold-worked specimen (Fig. 2a). Estimation of the magnetic phase content, before and after ageing treatment, in all the cold-worked specimens of AISI 304 stainless steel, indicated an increase in magnetic phase content after ageing in all the specimens.

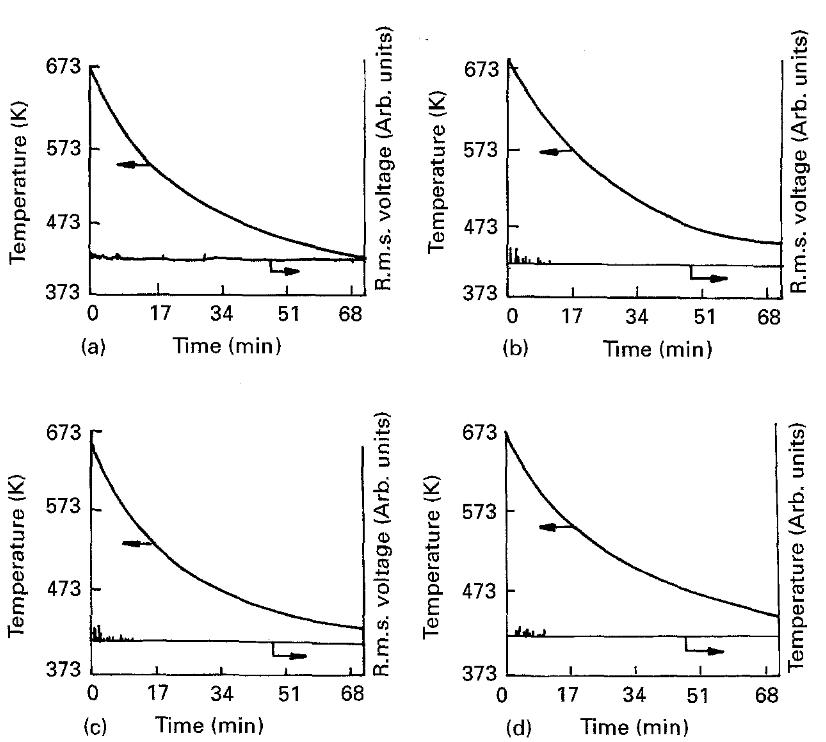

Figure 1 Variation of root mean square (r.m.s.) voltage of the AE signal and temperature with time during cooling of (a) the furnace without any specimen, (b) annealed AISI 316 stainless steel, (c) 50% cold-worked AISI 316 stainless steel, and (d) annealed AISI 304 stainless steel.

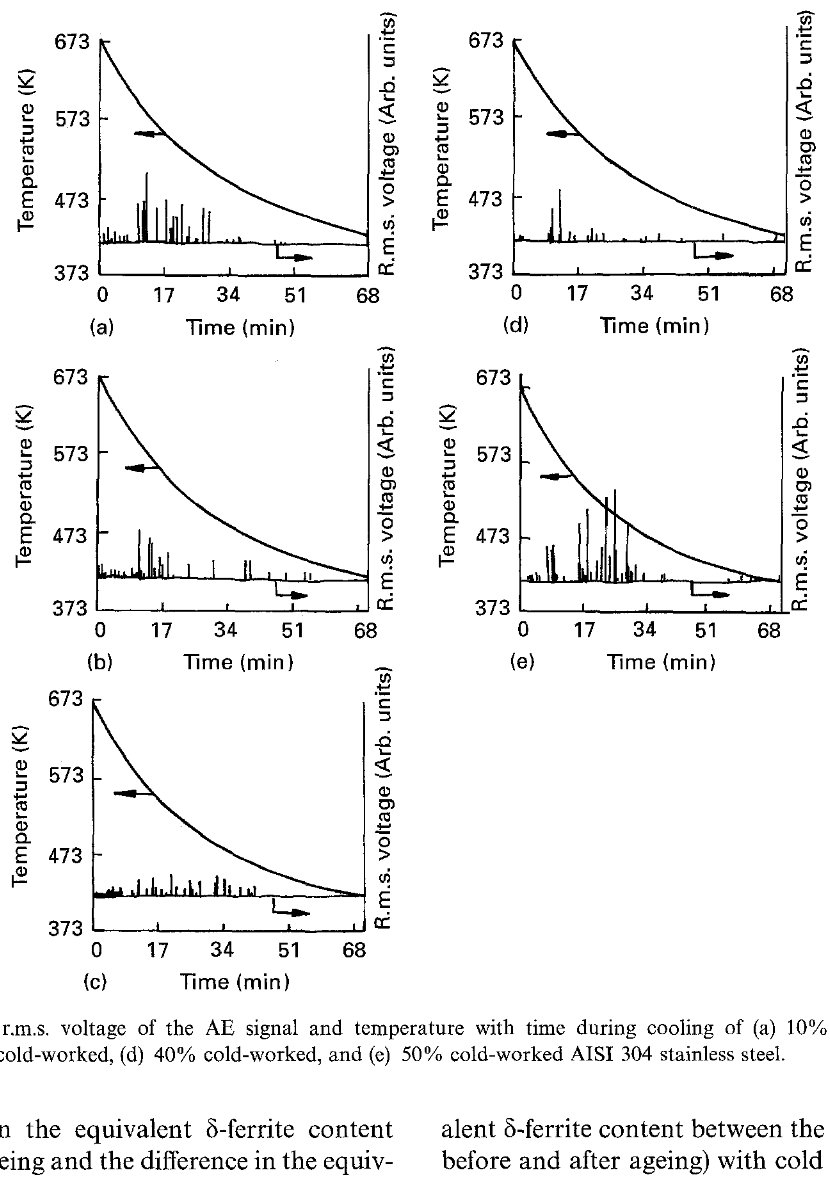

Figure 2 Variation of r.m.s. voltage of the AE signal and temperature with time during cooling of (a) 10% cold-worked, (b) 20% cold-worked, (c) 30% cold-worked, (d) 40% cold-worked, and (e) 50% cold-worked AISI 304 stainless steel.

The variation in the equivalent δ-ferrite content before and after ageing and the difference in the equivalent δ-ferrite content between the two conditions (i.e. before and after ageing) with cold work are shown in

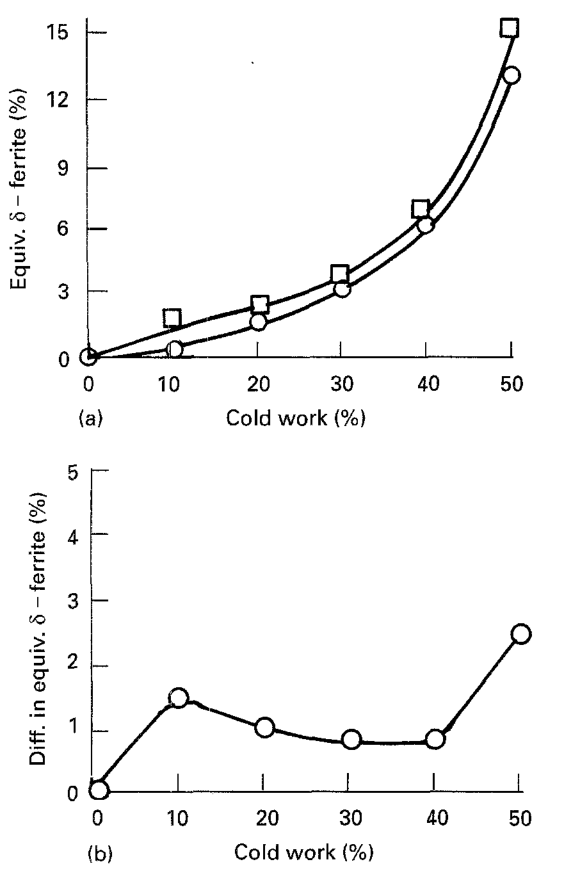

Figure 3 Variation of (a) equivalent δ-ferrite and (b) the difference in equivalent δ-ferrite (between (□) cold-worked and aged, and (○) as-cold-worked conditions) with prior cold work.

Fig. 3a and b. The latter plot is a measure of α′-martensite formed during cooling of aged specimens. It can be seen from Fig. 3b that the difference in the magnetic phase content before and after ageing is higher for 10% and 50% cold-worked specimens and lower for 20%–40% cold-worked specimens; in agreeing with the extent of acoustic emission generated in the cold-worked specimens. It is also seen that the increase in the amount of magnetic phase is greater in the 50% cold-worked specimen compared to that in the 10% cold-worked specimen, again supporting the higher acoustic emission generated in the 50% cold-worked specimen compared to the 10% cold-worked specimen.

As mentioned earlier, the formation of α′-martensite in cold-worked AISI type 304 stainless steel after ageing at temperatures below 873 K has been reported [11]. It is known that prior cold work enhances carbide precipitation in subsequent ageing [12]. The effect is much greater in the martensite containing AISI type 304 stainless steel than in AISI type 316 stainless steel. This is because of the fact that chromium and carbon can diffuse more rapidly in martensite (b c c) than in austenite (f c c) [13]. Moreover, carbon is also much less soluble in martensite than in austenite. Therefore, the driving force to form the carbides is greater in the presence of martensite [14]. This means that most of the carbon in the martensite rapidly goes into the formation of carbides, thus giving rise to a high density of fine carbides [15]. The higher dislocation density existing in the martensite also provides many favourable sites for carbide nucleation [11]. Therefore, with the presence of strain-induced α′-martensite in cold-worked AISI type 304 stainless steel, the chemical composition of the matrix at the sites of chromium and carbon depletion is altered during ageing, and this results in additional formation of α′-martensite upon cooling to the ambient temperature. Therefore, a substantial amount of AE generated in the cold-worked AISI 304 stainless steel specimens (Fig. 2a–e) in the temperature range 603–466 K during cooling, as compared to the negligible AE observed in other specimens (Fig. 1a–d) considered in this study is attributed to the formation of α′-martensite in the cold-worked 304 stainless steel specimens during cooling from the ageing temperature.

It has been already mentioned that a small amount of cold work stimulates the α′ formation, while extensive cold work suppresses the α′ formation during subsequent transformation by cooling [6, 7]. Similar behaviour was observed by the present authors in the cold-worked AISI 304 stainless steel specimens even when the tensile deformation was carried out at ambient temperature (~298 K) [16]. Simultaneous monitoring of AE during tensile deformation and measurements of magnetic phase content before and after tensile deformation have been used to establish conclusively this behaviour [16]. The higher amount of α′-martensite formation and the associated AE in the 10% cold-worked specimen is, therefore, attributed to the stimulating effect of a small amount of prior deformation for α′ formation during cooling after ageing. With increase in cold work, stabilization of the untransformed austenite takes place which suppresses subsequent nucleation [7]. At large deformation, the stabilizing effect of the dense dislocation tangles predominate over the stimulating effect of the stacking fault nuclei and the transformation is retarded [8]. The reduced amount of α′ formation for 20%–40% cold-worked specimens can therefore be attributed to the stabilization of austenite brought about by prior deformation.

Similar behaviour in the variation of α′ formation with cold work was reported after ausforming Fe–Ni–C alloy at 798 K [17]. Stimulation after smaller cold work and suppression after higher cold work was observed. It was also reported [17], that prior deformation of more than 40% enhanced α′ formation upon cooling. This was attributed to the increased fluctuation of the carbon concentration, leading to unstabilizing of the austenite and resulting in a higher amount of α′ formation during cooling. The higher amount of α′ formation in the 50% cold-worked specimens of the present study is, therefore, attributed to the similar unstabilization of the austenite.

The temperature range of α′ formation as detected by AET is the same for all the cold-worked specimens and lies between 603 and 466 K during cooling. This is not in agreement with the M_s temperature of 480 K reported by Butler and Burke [3] for initiation of α′ nucleation in annealed 304 stainless steel. This difference can be attributed to the different factors responsible for α′ nucleation, i.e. the available chemical driving force (altered chemical composition of the matrix) in the areas of chromium and carbon depletion, the interfacial energy requirements of good planar matching between austenite and martensite and the existence of favourable pockets of strain inhomogeneities [3]. Nucleation of α' phase during cooling depends on the local increase in M_s temperature which is different at different places for a given specimen and also varies in the different cold-worked specimens. This results in a range of temperature over which α' nucleation takes place. Chromium concentration increases with the distance from the precipitated zone and, correspondingly, the M_s temperature would have the opposite effect. This results in a temperature range for α' nucleation [3]. As mentioned earlier [5], generation of AE associated with α'-martensite formation also conclusively establishes that the formation of α' takes place by a shear process as suggested by Mangonon and Thomas [1] and not by a nucleation and growth process, as suggested by Chukhleb and Martynov [4].

4. Conclusion

AET has been used to study the α'-martensite formation in cold-worked AISI type 304 stainless steel during cooling after ageing for 1 h at 673 K. The temperature range of α' formation detected by AET lies between 603 and 466 K. A good correlation has been observed between the AE activity and the amount of α'-martensite formed in different cold-worked specimens. This variation could be explained based on the influence of the cold work and ageing on the stability of austenite.

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