Regular article

Electric current induced precipitation in maraging steel

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A B S T R A C T

Maraging steel of grade 350 (Fe-18Ni-11.6Co-3.6Mo-1.2Ti-0.13Al in wt%) was subjected to pulsed electric current (electropulsing) at current densities ranging from 4 to 10 kA/mm². Hardness increased with increasing current density in the solutionized condition. Electropulsing did not alter the grain size, but did increase the local misorientation within grains. Differential scanning calorimetry and atom probe tomography confirmed the precipitation of Ni₃(Ti, Mo). The temperature-time combination experienced by material due to joule heating was estimated to be insufficient for precipitation, and it was inferred that electric current inherently leads to an enhancement of precipitation kinetics.

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That the flow of electric current through a material alters its strength and microstructure has been known for over three decades [1–5], however the understanding of this phenomena is still not well established. In most studies the method of passing electric current through materials is by using current pulses of about 100 to 200 μs wide and with a magnitude so as to achieve a current density (J) through the material of 0.1 to 10 kA/mm². This process of passing electric current pulses through a material is referred to as electropulsing. Electropulsing carried out during deformation has been shown to reduce the flow stress [6–8] and has been attributed to the interaction of the conduction electrons with the elastic field of dislocations. Experiments have shown that electropulsing alters the mobility of the dislocations. Thus recovery, recrystallization and grain growth – all of which involve dislocation motion – would be affected by electric current [9–16]. Electropulsing has been seen to affect the morphology and distribution of carbides in steel [17–23] and the formation and dissolution of precipitates in Mg, Al and steels [24–30]. This phenomena of electric current induced phase transformations has been attributed to a decrease in the free energy of the transformation [31,32], thereby enabling the transformations to take place at lower temperatures and smaller times.

Maraging steel is a precipitation hardenable Fe-Ni bcc martensite steel which forms nanometer-sized Ni₃(Ti, Mo) precipitates upon aging [33–36]. The typical peak aging of maraging steel is done in the 490 to 510 °C range for about 4 to 6 h depending on aging temperature. The resulting precipitates are of tens of nm in size and are distributed in a martensite matrix having fine laths of about 50 to 100 nm width. As with any martensite, it has high dislocation density [37,38] and a large number of nanometer-sized precipitates, making it one of the strongest steels at ambient temperature. As seen in other materials, here too it is expected that electropulsing would lead to microstructural modification – such as either a break-up of the laths, a change in dislocation density or precipitation – possibly leading to further strengthening.

With this aim, maraging steel of grade 350 of composition 18Ni-11.6Co-3.6Mo-1.2Ti-0.13Al-0.004C (wt%) balance Fe, (abbreviated here as M350) in the solution annealed condition (820 °C for 1 h, here referred to as SA condition) was electropulsed using a circuit shown in Fig. 1(a). The thickness and length of M350 samples used for electropulsing were 1.1 and 56 mm, respectively, and the width ranged from 18 to 25 mm depending on the desired current density. Electropulsing of the SA samples was carried out at 9 different current densities (from 4.6 to 10 kA/mm²), achieved by varying the charging voltage (4 to 7 kV). The current density through the sample showed a damped oscillation characteristic (Fig. 1b) with the pulse lasting for about 200 μs. As a comparison with the SA condition, two samples of the solutionized and aged condition (490 °C for 5 h, referred to as aged) were also electropulsed.

Electropulsing of SA sample resulted in an increase in the Vickers microhardness (using 500 gf load) with increasing Jmax (maximum current density) as shown in Fig. 2. There was no change in hardness below Jmax = 5.6 kA/mm², and a significant increase in hardness beyond 8 kA/mm². In contrast, the hardness of the aged sample decreased with increasing Jmax. The increase in hardness of the SA condition and the decrease in the aged condition suggest that electropulsing could be the cause of precipitation and dissolution, respectively. It is also possible that electropulsing could lead to microstructural changes such as grain size and dislocation arrangements. To study this, electron backscatter diffraction (EBSD) was carried out in a FESEM using 20 kV operating...
The increased local strains observed after electropulsing could arise either due to an excess of geometrically necessary dislocations or due to the coherency strains of precipitation.

To confirm the occurrence of precipitation, differential scanning calorimetry (DSC) was carried out on SA, SA + 10 kA/mm² and aged +9 kA/mm² samples from 25 to 630 °C at a heating rate of 18 °C/min. Each sample was rescanned to obtain its baseline. As expected, the SA condition showed an exothermic peak from 200 to beyond 520 °C representing precipitation in these steels [36,39]. However, the DSC scan of SA + 10 kA/mm² showed no precipitation peak in the lower temperature range, implying that electropulsing resulted in early precipitation. Further, the aged sample after electropulsing showed a small peak at the lower temperatures (the as-aged sample showed no precipitation during a DSC scan). This suggests that electropulsing of the aged sample resulted in partial dissolution of the existing precipitates. In addition, atom probe tomography (APT) of SA + 10 kA/mm² sample (Fig. 4b) provided direct evidence for the precipitation of Ni₃(Ti, Mo)-type intermetallic phase (the known precipitate in maraging steel [33–36]). Detailed chemical composition of the precipitates, as determined by APT analysis is given in Table 1. These precipitates also showed considerable solubility of Fe, Co and Mo. Quite predictably, APT analysis of the SA sample did not show any such precipitation. The above results confirm that electropulsing of solution annealed M350 results in precipitation, whereas electropulsing of aged M350 resulted in partial dissolution of existing precipitates. To the best of the authors’ knowledge, precipitation due to electropulsing in maraging steel has not been reported earlier.

Precipitation in maraging steel reportedly occurs in the temperature range from 400 to 550 °C when held over for a few minutes to a few hours, depending on the temperature [33,34,40]. Some studies have shown that aging for less than a minute also results in initial precipitation in maraging steel, detected by an increase in hardness [40,41]. For example, aging maraging steel at 350 °C for 1 min increased the hardness from 340 HVN to 360 HVN, and aging at 500 °C for 1 min increased it to 520 HVN. In the present work too electropulsing of SA to 10 kJ/mm² resulted in a hardness of about 540 HVN (Fig. 2), suggesting that joule heating due to electropulsing could be the probable cause for precipitation. The joule heat goes to raising the sample temperature according to,

$$mC_p\Delta T = \int_0^t I^2Rdt,$$

(1)

where $m$ is mass, $C_p$ is specific heat capacity (taken as 500 kJ/g), $T$ is temperature, $I$ is current, $R$ is resistance and $t$ is the time. Integrating Eq. (1) over time gives the temperature rise $\Delta T$

$$\Delta T = \frac{\rho_e}{\rho_mC_p} \int_0^t J^2dt,$$

(2)

where $\rho_e$ is the mass density (8080 kg/m³), $\rho_e$ is the electrical resistivity (6.7 × 10⁻⁷ Ωm) and $J$ is the current density obtained from the experimental $J$-$t$ data (Fig. 1). The temperature rise due to electropulsing was also measured using a K-type thermocouple in contact with the sample surface. To prevent damage to the multimeter used to measure the temperature, the thermocouple was connected to the multimeter only after the electric pulse had passed through the sample. The delay in time from the end of the pulse to the measurement of temperature was within 5 s. The measured temperatures and that calculated from Eq. (2) are listed in Table 2. Note that at the highest current density the temperature was measured as 450 °C and was observed to drop below 200 °C within a minute. Given the 5 s delay in temperature measurement, the sample temperature just after the pulse was estimated (from simple 1-D heat transfer solution) to be about 500 °C, which is what was calculated from Eq. (2). In literature it was shown that hardness of 540 HVN in M350 can be achieved by the following alternative