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Strain-Ageing of Low-Alloyed Multiphase High-Strength Steels

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Abstract: The strain-aging of low alloyed, multiphase high-strength steels with strain-induced austenite to martensite transformation was studied. The influence of prestrain, aging time, and temperature dependence of the static strain aging was carried out. Ageing temperatures between 60 and 220 °C and aging times from 20 to 10,000 min were investigated. The choice of steel composition allowed studying the influence of alloying elements, such as Si and Al, on the static strain aging behavior. Samples after aging were studied using light-optical microscopy, X-ray diffraction, and in-depth transmission electron microscopy (TEM). The Harper model was used to describe the precipitation mechanisms occurring during aging. The study of thin foils after aging using TEM showed the precipitation of low temperature transition carbides in the microstructure, which was observed between 60 and 5000 min. By using X-ray diffraction, it was revealed that aging at 170 °C for a long time caused a slight decrease of the retained austenite volume fraction, but the C content remained constant.

Keywords: static strain aging; Advanced High-Strength Steels; bake-hardening; Cottrell and Bilby model; harper model; transmission electron microscopy

1. Introduction

Low-alloyed, multiphase high-strength steels exhibit superior mechanical properties and excellent crashworthiness for automotive applications. These steels allow carmakers to achieve considerable weight savings and to decrease fuel consumption, which allows important reductions of greenhouse gas emissions. Due to the importance of paint-baking in automotive manufacturing, the static strain aging phenomenon has gained considerable interest. The high-strength steels have a multiphase microstructure, consisting of ferrite, bainite, retained austenite, and martensite in various volume fraction. They are known as “Advanced High-Strength Steels” (AHSS). Static strain aging phenomena in steels depend on various factors, such as temperature, time, paint-baking, plastic deformation, and residual stress. Strain aging is associated with the long-range diffusion of interstitial atoms, such as C and N, to the dislocation strain fields. The aging process was originally described by Cottrell and Bilby [1]. Atmospheres are formed in the vicinity of the dislocation cores by diffusion of the interstitials. The initial stage of the aging process is described by the segregating total number of solute atoms $N(t)$. The equation for $N(t)$ is given by:

$$N(t) = n_0 \cdot \frac{\pi^{\frac{1}{3}}}{2} \cdot \frac{ADt^{\frac{2}{3}}}{kT} \quad (1)$$

where D is the diffusion coefficient of segregating solute atoms, T is the absolute temperature, t is the time, and the segregation is given per unit length of dislocation. n_0 corresponds to the solute atom number and n_0 is given per unit volume. A is the energy for the dislocation–solute atom interaction. Harper [2] modified Equation (1). In the proposed model, it is assumed that the segregation rate is proportional to the solute atom concentration. The model describes for a given time the fraction of atoms (W) which are already segregated:

$$\ln(1 - W) = -\frac{t}{\tau} = -(K \cdot t)^n \quad (2)$$

where K is a constant in relation with the enthalpy of activation and n is an exponent, which gives interpretation of the segregation mechanism. Low temperature transition carbides such as ϵ or η carbides may form during aging for intermediate time or at lower temperature [3]. It has been reported that the static strain aging process for a long time leads to the formation of carbides, such as θ in Fe-Mn-C alloys [4,5].

In the last decades, substantial work has been carried out to study the strain aging of ultra-low carbon (ULC) steels [6,7] and low-carbon (LC) steels [8]. Dual-phase (DP) steels [9,10] and transformation induced plasticity (TRIP) steels [11,12] have a multiphase microstructure. The static strain aging behavior of each constituent plays an important role. The constituents of the TRIP steels are ferrite, carbide free bainitic ferrite, and retained austenite. A specific two-step thermal treatment is used to obtain the TRIP microstructure. It consists of an intercritical annealing followed by isothermal holding at the temperature of austenite to bainite transformation [13]. Carbon enrichment plays a prominent role in setting the volume fractions of the different constituents and in the solid solution strengthening. Carbon enrichment of the retained austenite is realized in order to have the M_s^c temperature of 15–25 °C below room temperature [14]. Therefore, this allows the strain-induced austenite to martensite transformation to occur during deformation. The later contributes to enhance the ductility and to give superior strength level. In addition, bake-hardening increases the yield stress by aging (static strain-aging). The aging time of 20 min at 170 °C was studied, which corresponds to the industrial standard time applied during the paint-baking of automotive passenger cars [15,16]. Although the knowledge for fine tuning of the mechanical properties of the TRIP steels by means of an appropriate heat treatment are well developed, some issues regarding their additional strengthening by means of strain aging during bake-hardening remain unsolved. In the present contribution, the influence of prestrain, time, and temperature on the static strain aging behavior of several TRIP steels was investigated.

2. Materials and Experimental Procedure

The choice of materials made it possible to study different alloying compositions of conventional CMnSi, CMnSiAl, and CMnAl TRIP steels. The chemical composition of the TRIP steels is given in Table 1. A two-step heat treatment consisting of intercritical annealing and subsequent holding at the temperature of isothermal bainitic transformation was used to obtain the TRIP microstructure. The temperature to obtain the microstructure with 50 vol.% of ferrite and 50 vol.% of austenite during intercritical annealing was determined using Thermo-Calc simulations with the TCFE5 database. The time for intercritical annealing was 2 min. The time and temperature for holding during the isothermal bainitic transformation were 2 min and 460 °C, respectively. The steels were heat treated by means of a continuous annealing simulator (CASIM). The steels were investigated without temper rolling (TRIP1 to TRIP4), with the exception of the TRIP5 steel. TRIP5 steel was 1% temper rolled after the heat-treatment. The TRIP steels were characterized by light optical microscopy and the steels exhibited a typical microstructure with about 50 vol.-% of ferrite, 40 vol.-% of carbide-free bainitic ferrite, and 10 vol.-% of retained austenite. Tensile specimens according to the European Standard specification oriented parallel to the rolling direction were machined from the as-annealed material. The mechanical properties were determined by static tensile tests. The initial strain rate was $5.6 \times 10^{-3} \text{ s}^{-1}$

and it was increased after a strain of 3.4% to $5.6 \times 10^{-3} \text{ s}^{-1}$. During prestraining, the strain rate of $5.6 \times 10^{-3} \text{ s}^{-1}$ was kept constant. The investigated steels were prestrained in the range of 0–10% and aged in an oil bath at 60, 100, 170, and 220 °C for an aging time varying from 20 to 10,000 min. To study the bake-hardening process, the applied prestrains were conducted with 0%, 2%, 5%, and 10% tensile strain. European standard test specification was taken to determine the bake-hardening index BH_x , where x is the prestrain level. The retained austenite volume fraction was determined by means of X-ray diffraction (XRD). A filtered Mo- $K\alpha$ radiation ($\lambda = 0.070926 \text{ nm}$) was used. The method of Onink [17] was used to determine the C content of the retained austenite. This method of Onink et al. was taken, because it is well adapted for TRIP steels [14]. A transmission electron microscope (TEM) of type Philips EM420 TWIN-STEM was used to examine the microstructure using thin-foil. The steel samples were carefully polished and samples of 3 mm discs were prepared. The thin foils followed an electrochemical thinning at 16 °C with electrolytical polishing double-jet equipment. The A8 type solution containing 95 vol.-% of acetic acid and 5 vol.-% of perchloric acid from Struers was employed. The applied voltage was 32 V. The dislocation density measurement was determined by using the Ham's intercept method [18]. The two beam condition was used to image the dislocations. The foil thickness of the investigated region was obtained by using the procedure described by Kelly et al. [19]. Internal friction measurements were conducted using Automated Piezoelectric Ultrasonic Composite Oscillator Technique. The variable frequency internal friction measurements were carried out on the TRIP5 steel before and after the aging process. The bake-hardening temperature of the specimens for the internal friction was 170 °C and the aging time of 20 min. The internal friction specimens were machined in the rolling direction. The specimens were 58 mm in length, 3 mm in width, and 0.8 mm in thickness. The range of frequency was from $8 \times 10^{-4} \text{ Hz}$ to 10 Hz. The microstructures were studied at the K and L positions on the A_{80} specimens. The investigated BH conditions and labels are as follows:

- in the head of the tested samples of the as-received material, labeled AR-K;
- in the uniformly deformed part of the tested as-received material, labeled AR-L;
- in the head of the specimen 0% prestrain followed by bake-hardening at 170 °C for 20 min, labeled $BH_0\text{-K}$;
- in the uniformly deformed part of the tested 0% prestrain followed by bake-hardening at 170 °C for 20 min specimen, labeled $BH_2\text{-K}$;
- in the head of the specimen 2% prestrain followed by bake-hardening at 170 °C for 20 min, labeled $BH_0\text{-K}$; and
- in the uniformly deformed part of the tested 2% prestrain followed by bake-hardening at 170 °C for 20 min specimen, labeled $BH_2\text{-L}$.

Table 1. Chemical composition (wt%) of the CMnSi (TRIP1), CMnSiAl (TRIP2), CMnAl (TRIP3), CMnAl (TRIP4), and CMnSiAl (TRIP5) TRIP steels.

Steel	C	Si	Mn	P	Al
TRIP1	0.195	1.582	1.64	0.011	0.032
TRIP2	0.212	1.527	1.778	0.009	0.038
TRIP3	0.180	0.700	1.61	0.012	1.450
TRIP4	0.220	0.040	1.66	0.012	1.490
TRIP5	0.221	0.09	1.68	0.012	1.485

3. Results

3.1. Influence of the Prestrain

The influence of the prestrain level on the BH-behavior of typical CMnSi, CMnSiAl, and CMnAl TRIP steel grades (i.e., TRIP1–TRIP5) is shown in Figure 1. At a low temperature of 60 °C, low BH_0 -values in the range of 0–20 MPa were observed, as shown in Figure 1a. Slightly higher values

were observed for the TRIP2 steel, which remained higher than the other steels up to bake-hardening 10% prestrain. A maximum was reached for the 10% prestrained condition with BH_{10} values between 80 MPa and 100 MPa for the other TRIP steels. The BH_{10} value of 130 MPa was obtained for the TRIP2 steel. With increasing temperature up to 100 °C, the BH values for the investigated TRIP steels were slightly shifted towards higher prestrains (Figure 1b). The BH_0 values for the temperature of 60 °C decreased to lower values of 20–30 MPa. For the conventional BH-conditions of 170 °C, this effect was more pronounced, as shown in Figure 1c. Significant BH_0 were observed for the TRIP steels, which could reach of 50 MPa. BH_2 reached values up to 100 MPa. A further temperature increase to 220 °C improved the strength values of BH_2 to 100–120 MPa and BH_{10} to 170–190 MPa (Figure 1d).

Figure 1. Bake-hardening values versus prestrain for the investigated TRIP1–TRIP5 steels for 20 min at different aging temperatures (a) 60 °C, (b) 100 °C, (c) 170 °C, (d) 220 °C

3.2. Influence of the Bake-Hardening Temperature

The influence of the temperature on the bake-hardening values is shown in Figure 2. Increasing the temperature from 60 to 220 °C resulted in a significant increase of the BH_0 values (Figure 2a). BH_0 values in the range of 0–20 MPa were obtained for the temperatures of 60 and 100 °C. The sigmoidal shape of the curves for the investigated TRIP steel reached a maximum of about 220 °C. In Figure 2b, higher BH_2 values were obtained at 60 °C. For the temperature between 60 and 220 °C, the TRIP3 steel showed lower BH_2 values in the range of 20–50 MPa compared with the other TRIP steels. For

5% prestrain and 20 min aging, an increase of the BH values was observed (Figure 2c). High BH₅ values above 100 °C were observed. The TRIP4 steel had relatively high BH values up to 190 MPa in comparison with the other TRIP steels. High BH₁₀ values were in the range of 80–120 MPa for the temperature between 60 and 100 °C, as shown in Figure 2d. The BH values increased to relatively high values up to 160–180 MPa for 220 °C. The BH response was observed to be more important at higher temperature and higher prestrain level.

Figure 2. Bake-hardening values versus temperature for the investigated TRIP1–TRIP5 steels for 20 min of aging with different prestrain values for BH_x (a) BH₀ (with no prestrain), (b) BH₂ (2 % prestrain), (c) BH₅ (5 % prestrain), (d) BH₁₀ (10 % prestrain).

3.3. Influence of the Bake-Hardening Time

The influence of the aging time on the bake-hardening of the TRIP3 and TRIP5 steels was investigated for 5–10,000 min. The samples were prestrained to 0%, 2%, 5% and 10% and aged at a temperature of 170 °C (Figures 3 and 4). The bake-hardening values of the TRIP5 steel are shown in Figure 3. The results show high BH values for all investigated TRIP steels. The lowest BH value of 30 MPa was obtained for an unstrained sample after an aging time of 5 min. The highest values were obtained after 2% and 5% of prestrain. Significantly lower BH₁₀ values were obtained. BH₀ and BH₁₀ were in the same range for the aging times up to 100 min while the BH₁₀ values were significantly higher than the BH₀ values for longer aging times. Three aging stages could be distinguished. For the aging time from 5 to 1000 min, the BH values increased and then slightly decreased by ~20 MPa for

aging time of 10,000 min, as shown in Figure 5. For an aging time from 5 to 10,000 min, the BH values decreased significantly from 5% to 10% of prestrain. A first maximum is present at 60 min separating the first two stages of increasing BH values. A maximum of the yield point elongation (YPE) can also be seen at about this time in Figure 6. The third stage, starting at 1000 min, was characterized by decreasing BH values. By fitting the results obtained with the Harper model [2,20], one obtains in the first stage $t^{2/3}$ kinetics for BH_0 while for BH_2 , BH_5 , and BH_{10} a $t^{1/2}$ dependence is obtained. In the second and third stage, $t^{1/2}$ kinetics were found for the unstrained and all the prestrained samples. The evolution of the tensile stress is given in Figure 7. The tensile stress of the 5% and 10% prestrained samples is higher than the tensile stress of the base material even at aging times below 100 min. For the 2% prestrained samples, the tensile stress raised above the original tensile stress in the second and third stage of aging. It is noteworthy that the high BH values of the 10% prestrained samples did not decrease for longer aging times.

For the TRIP3 steel, the BH_0 values were in the range of 5–33 MPa (Figure 4). The BH_2 values were rather similar than the ones of the BH_0 values for an aging time less than 60 min. They increased significantly for longer aging time. Two maxima for the BH_0 and BH_2 values were observed. The first one was for an aging time of 60 min and the second one for 5000 min. Based on these results, one can assume that the aging time of 60 min corresponds to the end of the Cottrell formation. The first maximum separates the first two stages of increasing BH values. The third stage, which starts at 5000 min, is characterized by the decrease of the BH values.

Between 5 and 10,000 min aging time, the BH values increased significantly after 2% of prestrain, as shown in Figure 8. Contrary to the TRIP5 steel, the BH_5 and BH_{10} values were rather similar for the same aging time in the range of 5–10,000 min. Figure 9 shows the evolution of yield point elongation as a function of aging time. For BH_0 , the YPE was constant for 60–100 min and decreased for an aging time longer than 5000 min. No maximum of YPE was observed for 60 min for BH_0 . BH_5 and BH_{10} exhibited relevant values compared to BH_0 and BH_2 . No maximum was observed for BH_5 and BH_{10} . The BH_5 and BH_{10} values were ~ 40 MPa after 10 min and increased to ~ 95 MPa after 5000 min. The BH values for 5% prestrain were slightly lower than the BH values for the 10% prestrain. This is related to the increased dislocation density with an increased level of prestrain. As shown in Figure 10, the tensile strength after aging for BH_0 , BH_5 , and BH_{10} increased with the aging time. From 1000 to 10,000 min, the tensile strength for the BH_2 values decreased by ~ 20 MPa.

Figure 3. BH kinetics at 170 °C from 5 to 10,000 min for 0%, 2%, 5%, and 5% prestrain for the temper rolled TRIP5 steel.

Figure 4. BH kinetics for aging at 170 °C from 5 to 10,000 min for 0%, 2%, 5%, and 5% prestrain for the TRIP3 steel.

Figure 5. BH values versus prestrain for aging at 170 °C from 5 to 10,000 min for the temper rolled TRIP5 steel.

Figure 6. Yield point elongation as function of the aging time for aging at 170 °C from 5 to 10,000 min for 0%, 2%, 5%, and 5% prestrain for the temper rolled TRIP5 steel.

Figure 7. Tensile strength after aging as function of the aging time for aging at 170 °C from 5 to 10,000 min for 0%, 2%, 5%, and 5% prestrain for the temper rolled TRIP5 steel.

Figure 8. BH values versus 0%, 2%, 5%, and 5% prestrain for aging at 170 °C from 5 to 10,000 min for the TRIP3 steel.

Figure 9. Yield point elongation as function of the aging time for aging at 170 °C from 5 to 10,000 min for 0%, 2%, 5%, and 5% prestrain for the TRIP3 steel.

Figure 10. Tensile strength after aging as function of the aging time for aging at 170 °C from 5 to 10,000 min for 0%, 2%, 5%, and 5% prestrain for the TRIP3 steel.

3.4. Retained Austenite Stability

Figure 11 shows the XRD diffractogram for the AR-K, AR-L, BH₀-K, BH₀-L, BH₂-K, and BH₂-L specimens of TRIP5 steel. Increasing the level of prestrain up to 2% resulted in the decrease of the retained austenite volume fraction. The effect of the prestrain in Figure 11 is visible especially by the decrease of the intensity of the (220)_γ and (311)_γ peaks. After bake-hardening for 20 min at 170 °C, no clear peak decrease and 2θ-shift were observed, which would be related to decomposition of the retained austenite and carbide formation. The influence of C content is shown in Table 2. This also shows that the retained austenite is still present in the steel microstructure.

Figure 12 shows the influence of BH₀ and BH₂ on the peaks of *bcc* and *fcc* for the TRIP5 steel. The (220)_γ peaks did not change with the influence of bake-hardening. However, the (311)_γ peak was slightly changed, which is probably due to straining and some influence on texture. The peaks of the ferrite and bainitic ferrite constituents for (200)_α and (211)_α did change after both bake-hardening BH₀ and BH₂. In Figure 13, a small change of the carbon content is visible after bake-hardening at 170 °C for 20 min.

Figure 14 shows the influence of aging time from 5 to 5000 min at 170 °C for the TRIP5 steel. No clear changes were observed regarding the intensity of the *bcc* and *fcc* peaks. The peaks corresponding to (200)_γ, (220)_γ, and (311)_γ did not change significantly with increasing aging time. Figure 15 shows the decrease of the retained austenite volume fraction as a function of the ageing time up to 5000 min for the TRIP5 steel. The C content in the retained austenite is also shown in the Figure. This confirms the stability of the retained austenite for long ageing time. It is noteworthy that this does not exclude the possible formation of low temperature transition carbides in the microstructure after a prolonged aging time (e.g., 5000 min).

Figure 11. X-ray diffraction diffractogram for the TRIP5 steel for the non-strained (K) and strained (L) samples without and with paint-baking at 170 °C for 20 min.

Figure 12. X-ray diffraction diffractogram showing the effect of the bake-hardening and prestraining for the TRIP5 steel (BH₀, paint-baking at 170 °C/20 min; BH₂, 2%prestrain, paint-baking at 170 °C/20 min).

Figure 13. Evolution of the carbon content and retained austenite in the different samples mybluefor the TRIP5 steel.

Figure 14. XRD diffractogram for the TRIP5 steel after aging at 170 °C for 5, 10, 60, 500, and 5000 min.

Figure 15. Evolution of the carbon content and volume fraction of retained austenite by means of XRD for the TRIP5 steel after aging at 170 °C for 5, 10, 60, 500, and 5000 min.

Table 2. XRD results before and after aging. The paint-baking simulations were at 170 °C for 20 min for the TRIP5 steel.

Steel	$V_{\gamma \text{ ret.}}, \%$	$C_{\gamma \text{ ret.}}, \text{wt}\%$
TRIP5, before aging	11.5	1.81
TRIP5, after aging	12.1	1.84

Similar results were obtained for the TRIP2 steel. No pronounced shift of 2θ was measured after bake-hardening at 170 °C for 20 min, as shown in Figure 16.

Figure 16. Evolution of the carbon content and volume fraction of retained austenite for the TRIP2 steel in the samples before and after aging at 170 °C for 20 min.

3.5. Internal Friction Measurement

ULC steels and LC steels are known to exhibit a Snoek peak [21–28] due to the very low amount of interstitials. Figure 17 shows the internal friction measurement for prestrain levels in the range of 0–2% of the TRIP5 steel. No clear Snoek-type peak was observed, as shown in the figure. In comparison with the prestrained samples, the higher damping at low frequency for the TRIP5 steel suggests the presence of a higher density of defects and more mobile dislocations. Increasing the prestrain up to 2% decreased the damping at low frequency. This is due to less mobile dislocations. Figure 18 shows the stress–strain curve of the TRIP5 steel. No clear discontinuous yielding was observed. The decrease of damping may be due to the contribution of the ferrite phase. Figure 19 shows the effect of the aging at 170 °C for 20 min for the 1% prestrained TRIP5 steel. For a low frequency, the damping decreased due to more dislocation pinning. No Snoek peak related to interstitial C was observed. Variable frequency internal friction measurements were carried out on the TRIP5 steel before and after BH. The bake-hardening temperature was 170 °C and the aging time of 20 min. No clear evidence of Snoek-type peak, related to interstitial C, was observed.

With the increase in the dislocation density, the magnitude of damping increases. The amplitude dependent damping results from the hysteresis phenomenon involving dislocation breaking away from their pinning points. However, with the increase of prestrain, the length of the dislocations are reduced, due to the dislocation–dislocation interactions [28].

Figure 17. Internal friction measurement for the TRIP5 steel with the prestrains in the range of 0–2%.

Figure 18. Stress–Strain curves for the TRIP5 steel without and with aging at 170 °C for 20 min.

Figure 19. Internal friction measurement for the TRIP5 steel with 1 % prestrain and after aging at 170 °C for 20 min.

3.6. Transmission Electron Microscopy

Transmission electron microscopy was carried out on the TRIP5 steel [14]. The microstructure revealed low dislocation density of $24 \times 10^9 \text{ cm}^{-2}$ in the polygonal ferrite. Bainitic ferrite was observed as lath-like type carbide free structure with high dislocation density using TEM. Retained austenite was observed more often in the vicinity of the grain boundaries or close to bainitic ferrite and ferrite.

Some pile up of dislocations were observed in the polygonal ferrite, usually from the edge of bainitic ferrite laths. After 2% and 5% prestrain, the observed main features are as follows: (a) a high dislocation density in the austenite; (b) heavily dislocated martensite without transformation twins; and (c) some evidence for deformation microtwinning. These results are shown in Figure 20. The microtwins were found to fully cross the retained austenite particles. The strain-induced martensite was mostly present as broad irregular bands crossing the retained austenite grains. Some austenite grains were almost fully transformed to martensite. Retained austenite grains, which did not contain either microtwins or martensite, had a high dislocation density. The generation of dislocation fronts was observed in the polygonal ferrite from the partially transformed retained austenite grains. Cell substructure formation was shown after straining, and particularly pronounced for higher prestrain, i.e., 10%.

After aging at 170 °C for 5 min, no clear carbides precipitation was observed in the ferritic matrix, bainitic ferrite, and retained austenite. However, for an intermediate ageing time, low temperature transition carbides were observed by TEM in the microstructure for an aging time less than 5000 min. The TEM study showed the precipitation of low temperature transition carbides mostly for an aging time between 60 and 5000 min. Low temperature transition carbides in the bainitic ferrite laths were observed, which is related to the higher C content of the bainitic constituent. The results showed low temperature transition ϵ or η carbides for an intermediate ageing time, which were located mostly within the laths of carbide free bainite. As shown in Figure 20(up)), the formation of carbides occurred in bainitic ferrite. Cementite formation was observed in the mostly in the bainitic ferrite laths after

aging at 170 °C for 5000 min. For a longer ageing time, the precipitation of cementite in the ferrite was more significantly observed. The fine carbide precipitation of cementite was clearly observed in the polygonal ferrite, in particular for long aging times of 5000 and 10,000 min (Figure 20(down)).

For 0% prestrain, the dislocation density was observed to slightly decrease with the increasing aging time. For aging at 170 °C for 5000 min, the dislocation density was determined to be $12 \times 10^9 \text{ cm}^{-2}$ and it remained unchanged for longer aging time up to 10,000 min. For 0% prestrain, the retained austenite remained stable up to 5000 min. It showed no indications of austenite decomposition. The XRD results show that the retained austenite is particularly stable at an aging temperature of 170 °C for an aging time up to 5000 min. At aging temperature of 170 °C, no clear influence of aging treatment on the volume fraction of retained austenite was observed.

Figure 20. TEM micrograph of the TRIP5 steel for the 2% prestrained and aged at 170 °C for 60 min, which reveals carbides in the structure of bainitic ferrite (up) and - the non-strained TRIP5 aged at 170 °C for longer ageing time of 5000 min with θ -carbide precipitation (down).

3.7. Discussion

The replacement of Si by Al causes a slowdown of the transformation kinetics. At higher prestrain, e.g., 5% or 10%, this can explain the difference of BH, because of less martensite in the microstructure of the TRIP3 steel. The higher is the prestrain, the higher is the dislocation density. The higher dislocation density may contribute to the higher yield strength. Due to the increased dislocation density of the bainitic constituent and increased C content [15,29], it is probable that the bainite contributes to a higher BH response than ferrite. Another effect can be entailed by the intrinsic transformation mechanism of the retained austenite changing to martensite under external stresses and thus providing for the strength increase visible in the BH-values. Considering the BH-values in dependency of the BH-temperature, the strength increase is more pronounced with higher temperatures. Within the relevant time range up to 20 min, the strengthening effect is related to the Cottrell-effect with dislocations being locked by the segregation of interstitial atoms in the stress field of the dislocations due to diffusion. Increasing the temperature of aging favors the diffusion of the interstitial atoms to the dislocations, which results in a greater bake-hardening response. More importantly, the bake-hardening response results from the composite effect of the constituents of TRIP steel. The ferrite requires the increase of prestrain in order to have higher dislocation density in the matrix, which promotes Cottrell effect. The carbide free bainitic constituent is characterized by an increased dislocation density, which offers an advantageous higher BH response.

No clear influence is observed on the volume fraction of retained austenite and the C content for the TRIP5 steel after aging for 5–10,000 min at 170 °C based on XRD-results. This confirms that the retained austenite is stable against decomposition. Considering the BH₅ and BH₁₀, no precipitation stage is observed for the TRIP3 steel. This results from the effect of the chemical composition on the austenite to martensite transformation kinetics in TRIP steel.

4. Conclusions

The bake-hardening kinetics were investigated for aging times in the range of 5–10,000 min for different TRIP steel compositions. For BH₀ to BH₂, both TRIP3 and TRIP5 steels exhibited the transition from the first stage to the second stage of aging for 60 min. No clear effect of the chemical composition was observed on the aging time transition. The BH₀ values can be explained by the locking of the mobile dislocations in the bainite constituent. Notice that the t^2 and t^3 kinetics of the BH₀ values of the TRIP5 steel suggest a minor formation of carbides.

X-ray diffraction measurements on the TRIP steels showed the influence of ageing time and temperature on the volume fraction and C content of retained austenite. The TEM study using thin-foils revealed the precipitation of low temperature transition carbides in the microstructure for an aging time between 60 and 5000 min. Longeraeaging resulted in the precipitation of cementite in the microstructure, especially in the ferrite and bainite constituents of the investigated TRIP steel. The TEM results showed the clear precipitation of cementite for a long aging time of 10,000 min.

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