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Strengthening phenomenon in 0.5%C steel hot-forged and subsequently held in stable austenite region

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0.5% C steel has been studied to clarify the correlation of mechanical properties and microstructure in steels hot-forged and subsequently held in the stable austenite region. Hot-forging at 900 or 1200 °C followed by intermediate holding at 800 or 1100 °C, respectively, prior to water quenching has been employed. Hardness and bend strength decreased with increasing intermediate holding time up to about 50 s. The subsequent intermediate holding produced a surprising and hitherto undescribed phenomenon because an abrupt strengthening occurred at an intermediate holding of 50 s, whereas previous austenite grain size gradually increased with intermediate holding time. Hence there was little difference between the mechanical properties of the steels with no intentional holding time (2 s) and a holding time of 50 s. The concurrent appearance of microtwinning in martensite, and a simultaneous decrease in the activation energy values of the first and third stages in tempering, were seen without an extreme change in retained austenite contents. The mechanism responsible for the strengthening phenomenon is discussed in terms of a carbon segregation model, in which carbon segregates to defects introduced into the austenite during hot-forging, followed by an intermediate holding prior to quenching.

1. Introduction

The mechanical properties of most alloys and steels are determined by their microstructure, which in many instances is controlled by thermal and thermomechanical treatments. However, conventional heat treatment of alloy steels cannot always meet current engineering requirements for improvements in strength without sacrificing ductility and toughness. The realm of thermomechanical treatments appears very attractive for developing the strength of low-alloy steels without sacrificing ductility and toughness [1, 2]. Particularly noticeable is the application of a high-temperature thermomechanical treatment (HTMT) at temperatures above Ac_3 (stable austenitic region) prior to quenching to form martensite. Therefore, this technique has been applied to low- and medium-alloy steels, in which metastable austenite cannot be retained below the temperature regime of stable austenite long enough to permit deformation. The strength increase obtained by HTMT processing is not as great as that of austenite forming, but ductility and toughness may be significantly increased. Recently Tomita [3] has investigated the effect of microstructure on the mechanical properties of 0.5 %C steel processed by HTMT. He has shown that HTMT resulted in a large improvement in toughness with a moderate increase in strength. However, since the deformation temperature is above the recrystallisation temperature, the mechanical properties resulting from HTMT are controlled by microstructural changes (grain growth of austenite) or substructural changes occurring after hot-forging [4–7]. Thus for good control of mechanical properties of steels processed by HTMT it is necessary to clarify the correlation of mechanical properties and microstructures of steels hot-forged and subsequently held in the stable austenite region.

In this work, 0.5 %C steel has been studied to clarify the correlation of mechanical properties and microstructure in steels hot-forged and subsequently held in the stable austenite region.

2. Experimental procedure

Commercial 0.5 %C steel, which was air-melted and vacuum-degassed, was used in this investigation. The chemical composition and Ar_1, Ar_3, and Ms temperatures are given in Table I. The steel was received as 80-mm-diameter hot-rolled bar stock. Test specimens were machined from the bars to 15 × 15 × 120 mm. Each was fully annealed.

The thermomechanical treatment schedule for the test specimens are given in Table II. Austenitization and intermediate holding were conducted using an argon atmosphere furnace. A 50 % reduction in thickness was made by one stroke at a strain rate of $80\ s^{-1}$ using an air hammer at maximum capacity of 1.47 MN. The time interval between the upsetting and

TABLE I Chemical composition of steel used (wt %)

C	Si	Mn	P	S
0.50	0.31	0.70	0.021	0.012

Ar_1, Ar_3 and Ms were determined by standard dilatometry: the values are, respectively, 721, 753 and 328 °C.

TABLE II Thermomechanical treatment schedule

Designation	Thermomechanical treatments
900HF–800IH–WQ	Austenitize at 900 °C (72 h), 50% hot-forge at one time and intermediate hold at 800 °C for required time, water quench.
1200HF–1100IH–WQ	Austenitize at 1200 °C (72 h), 50% hot-forge at one time and intermediate hold at 1100 °C for required time, water quench.

quenching for specimens with no intentional holding time was 2 s. After forging, the steels were held at intermediate temperatures, that is, finishing temperatures of forging, for the required time. The temperatures were measured by connecting a thermocouple wire spark-welded to the ends of the specimen, chromel to one side and alumel to the other side, to a high speed recorder. Specimens were quenched in water after holding at an intermediate temperature for the required time. Tempering was done in an oil bath at 150 °C for 1 h.

Mechanical properties were determined by Vickers Hardness (tested at 30 kg) and a three-point slow-bend test. The slow bend test (length of span = 50 mm) was carried out using specimens with dimensions of 4 × 20 × 80 mm on an Instron machine, at a cross-head speed of 0.01 mm s^{-1}, at room temperature. Bend strength was evaluated by fracture stress. The microstructure of the specimens was examined using optical, thin-foil transmission electron microscopy (TEM) and scanning electron microscopy (SEM). Previous austenite grain boundaries were delineated by first etching in a supersaturated aqueous solution of picric acid containing 0.2 wt % of a wetting agent [8, 9]. The prior austenite grain size was determined by ASTM linear analysis on optical micrographs. Thin foils were prepared by grinding to 0.1 mm thickness, then chemically thinning in a mixed solution of hydrofluoric acid and hydrogen peroxide, followed by electropolishing in a mixed solution of phosphoric and chromic acids. Retained austenite contents were determined by the X-ray technique developed by Miller [10] in which the sample surface is rotated and tilted to an incident beam using Mo$K\alpha$ radiation. A scanning speed of 0.0003 deg s^{-1} was generally used for weak austenite diffraction, compared with the through scanning speed normally employed for martensitic peaks of 0.003 deg s^{-1}. The combination of peaks chosen for the analysis was $(2\,1\,1)\alpha$, $(2\,2\,0)\gamma$, and $(3\,1\,1)\gamma$. Tempering kinetics were analysed using non-isothermal dilatometry [11]. The dilatometry was performed by continuous heating under vacuum at a controlled rate of 0.008 °C s^{-1}. The test specimens were 4 mm in diameter and 80 ± 0.25 mm long. The apparent activation energy, E, was evaluated by

$$E = -nRT_i^2 C_i^{n-1}(dC/dT)_i/C_i^n \qquad (1)$$

where n and R are, respectively, the order of reaction and the gas constant, and T_i, C_i and $(dC/dT)_i$ are the temperature, length fraction of reactant remaining, and the slope of the tangent at the inflection point of the curve plotted as the length reaction of reactant remaining against temperature, respectively.

3. Results

3.1. Mechanical tests

Fig. 1 shows the effect of intermediate holding time on the hardness of specimens processed by hot-forging at 900 °C followed by intermediate holding at 800 °C for the required time before water quenching (designated 900HF–800IH–WQ), and those processed by hot-forging at 1200 °C followed by intermediate holding at 1100 °C for the required time before water quenching (designated 1200HF–1100IH–WQ). As seen from this figure, the hardness decreased with increasing intermediate holding time up to about 50 s independent of forging or intermediate temperature. However, subsequent intermediate holding revealed a surprising and hitherto undescribed phenomenon of an abrupt increase in hardness at a holding time of 50 s. The phenomenon was more prominent for the 1200HF–1100IH–WQ specimens than for the 900HF–800IH–WQ specimens. The effect of intermediate holding time on the bend strength of the 900HF–800IH–WQ and 1200HF–1100IH–WQ specimens tempered at 150 °C for 1 h was investigated. The results are shown in Fig. 2. As seen from this figure, the same strengthening effect was observed at a holding time of 50 s.

3.2. Metallography and X-ray measurements

In order to clarify the mechanism responsible for the abrupt strengthening phenomenon observed during intermediate holding, metallography was carried out. Since the strengthening effect was most marked for the 1200HF–1100IH–WQ specimen, metallography was focused on that specimen. Fig. 3 shows the effect of intermediate holding time on the prior austenite grain size. As seen from this figure, the prior austenite grain size gradually increased with intermediate holding time. This suggests that the observed strengthening phenomenon is not correlated with changes in the prior austenite grain size. The results of TEM observations are shown in Fig. 4. The TEM observations revealed that the microstructure of steel with no intentional holding time consisted predominantly of subcell structure (Fig. 4a). The TEM observations also revealed that the subcell structure changed to subgrains as the intermediate holding time increased (Fig. 4b). The TEM observations revealed that microtwinning appeared in the subgrains at the intermediate holding

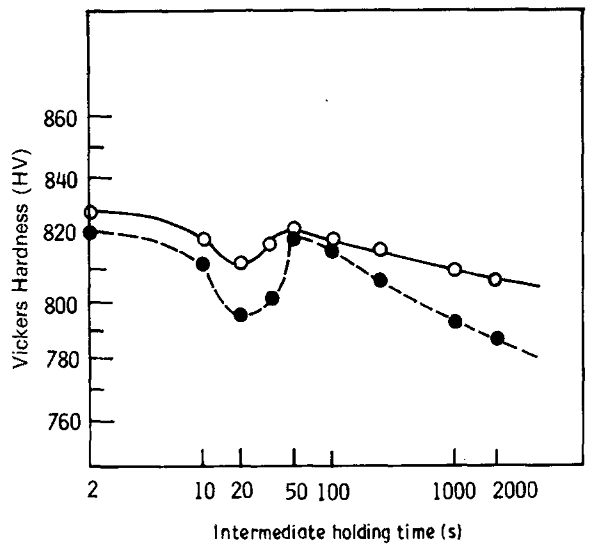


Figure 1 Effect of intermediate holding time on hardness of ○, 900HF-800IH-WQ and ●, 1200HF-1100IH-WQ specimens.

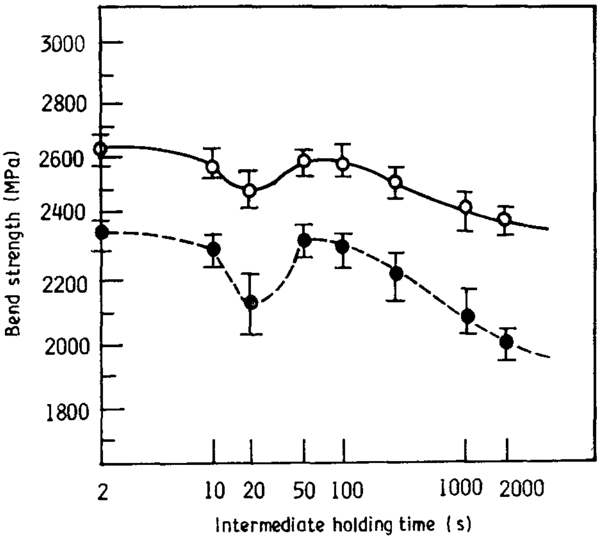


Figure 2 Effect of intermediate holding time on bend strength of ○, 900HF-800IH-WQ and ●, 1200HF-1100IH-WQ specimens tempered at 150 °C for 1 h.

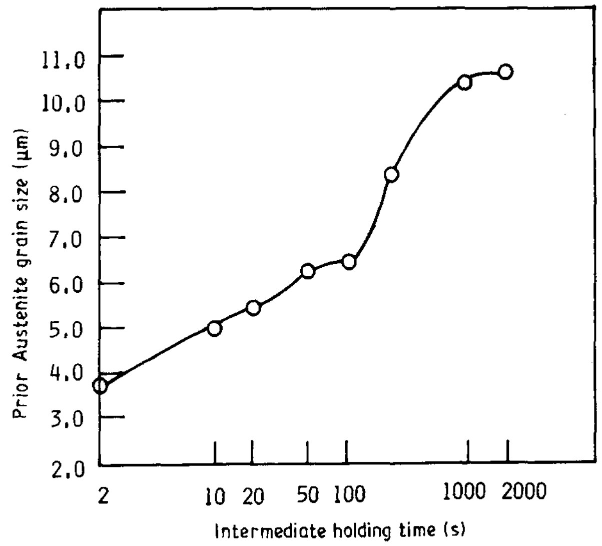


Figure 3 Effect of intermediate holding time on the prior austenite grain size of 1200HF-1100IH-WQ specimen.

time of 50 s (arrowed in Fig. 4c), this being concurrent with the observed strengthening phenomenon.

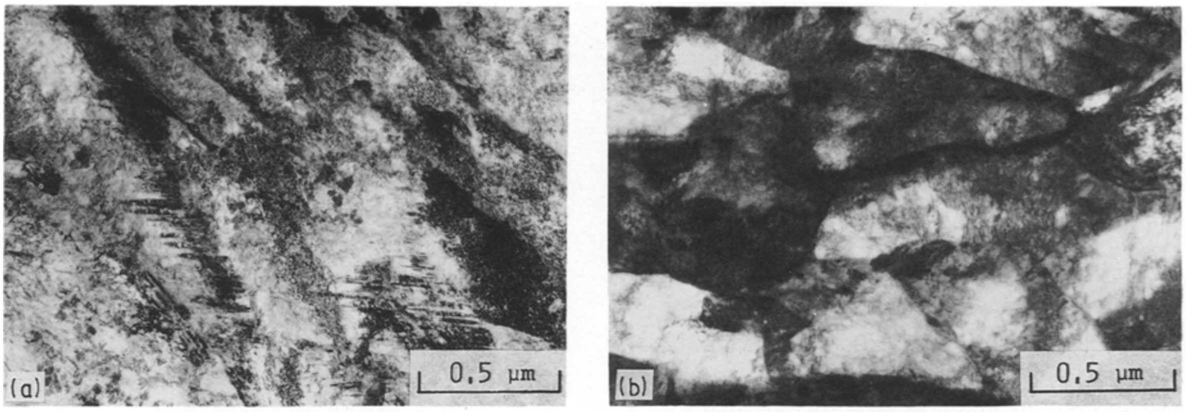
Retained austenite plays an important role in the mechanical properties of heat-treated low-alloy steels. The X-ray measurements revealed that the retained austenite contents increased from 3.2 to 4.0 vol % with increasing intermediate holding, but the difference was not prominent.

3.3. Non-isothermal dilatometry

In order to investigate further the mechanism of the strengthening effect occurred during holding at the intermediate temperature, tempering kinetics were determined by non-isothermal dilatometry. Fig. 5 shows a comparison of the temperature derivative of the dilation curves of the steels with intermediate holding times. The dilatometry revealed that temperatures at which the first and second stages begin increased when the intermediate holding time increased from 2 to 20 s. However, the dilatometry revealed that these temperatures decreased at a holding time of 50 s. To determine this effect quantitatively, the apparent activation energy values of the third stage of tempering were evaluated. The results for the 1200HF-1100IH-WQ specimen calculated using Equation 1 are shown in Table III. As seen from the table, the apparent activation energy value increased from 61.20 to 68.20 kJ mol^{-1} as the intermediate holding time increased from 2 to 20 s. However, the activation energy value decreased to 64.98 kJ mol^{-1} at the intermediate holding time of 50 s, while it had a tendency to increase again over a holding time of 100–200 s.

4. Discussion

It may be seen from the above data that the presence of microtwinning in martensite and decreased activation energy values of the first and third stages of tempering, are associated with the abrupt strengthening phenomenon observed following the intermediate holding. Because the microtwinning has a strengthening effect on steel possessing a lath morphology [12], this may permit an explanation for the strengthening phenomenon. Kelly and Nutting [12] have shown that microtwinning in martensite is encouraged at high carbon contents, while low-carbon martensite consists of a lath structure with no trace of microtwinning. A carbon segregation model is proposed in which, during the intermediate holding, carbon segregates locally to defects introduced in the austenite by hot-forging. Ansell and Arrot [13] have suggested that part of the strengthening behaviour of ferrous martensite in plain carbon and low alloy steels can be explained by the segregation of carbon in austenite during cooling prior to the martensite transformation. Ansell and Breinan [14] have also shown that as the austenite cools during quenching, the equilibrium concentration of carbon at defects (such as dislocations), in austenite increases. Furthermore, they have shown that when the austenite transforms to martensite, the carbon-rich areas are inherited in the martensitic structure. Thus, Ansell and coworkers [13,



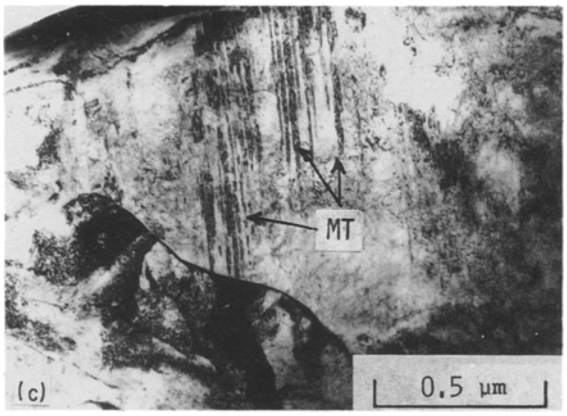


Figure 4 TEM micrographs of 1200HF–1100IH–WQ specimens. Specimens with (a) no intentional holding time (2 s); (b) 20 s holding times; (c) 50 s holding times. MT indicates microtwinning.

TABLE III Activation energy of third stage in tempering of 1200HF–1100IH–WQ specimens calculated using Equation 1 (heating rate = 0.008 °C s^{-1})*

Intermediate holding time (s)	T_i (K)	C_i (K^{-1})	$(dC/dT)_i$	Activation energy E (kJ mol^{-1})
2	523	0.39	-1.03×10^{-2}	61.20
20	545	0.38	-1.05×10^{-2}	68.20
50	530	0.37	-1.03×10^{-2}	64.98
100	550	0.38	-1.08×10^{-2}	71.44
200	571	0.37	-1.11×10^{-2}	81.28

*$n = 1$, $R = 8.31$ J mol K^{-1}.

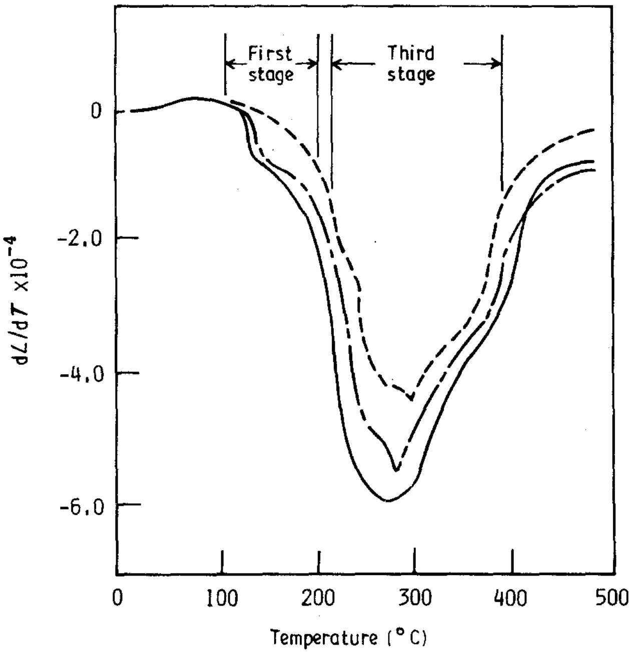


Figure 5 Temperature derivatives dL/dT against tempering temperature in dilation curves for 1200HF–1100IH–WQ (heating rate = 0.008 °C s^{-1}). Intermediate holding times: (——) 2 s; (- - -) 20 s; (— — —) 50 s.

14] have shown that the kinetics of the formation of these carbon-rich regions within the austenite during quenching should follow Equation 2 [15]

$$F = 1 - \exp\{-3\rho[\Delta V(\mu b/3\pi)(1 + \nu)/(1 - \nu)\, D(t/kT)]^{2/3}\} \quad (2)$$

where F is the fraction of the original solute segregated to dislocations in time t; ρ is the dislocation density; ΔV is the volume change due to the addition of one carbon atom to the lattice; b is the Burgers vector of the dislocation; ν is Poisson's ratio; k is Boltzmann constant; D is the diffusion rate of carbon in austenite; μ is the shear modulus; and T is the absolute temperature. This equation shows that if the steel has many defects and a finite holding time at intermediate temperature prior to quenching, local carbon segregation to defects increases even at high temperature. Since many defects appear in the austenite during hot-forging, and subsequently the austenite cools from 1200 to 1100 °C or 900 to 800 °C, followed by holding for required times at the intermediate temperatures, local carbon segregation to defects occurs and produces local microtwinning on quenching. Subsequently the carbon segregation will facilitate carbide precipitation during tempering. A question arises as to why the observed phenomenon is more prominent for the 1200HF–1100IH–WQ condition than for the 900HF–800IH–WQ condition. This could be due to the fact that the carbon segregation effect is changed by the forging and intermediate temperatures. However, further studies are required using different materials before any final conclusions concerning the mechanism can be drawn. In addition to the segregation of carbon, the segregation of different elements, in particular manganese, to austenite defects, should be considered. Manganese segregation gives rise to a local change in the stacking-fault energy of austenite which has been shown to exert a strong influence on the transformation characteristics on quenching, especially on the amount of twinning [16–19]. Ray and Seal [19] have shown the dramatic effect of an increase in manganese content on the amount of microtwinning in low-carbon steel. In the present investigation, it is possible that dissolution of manganese–sulphide inclusions could provide a source of high local concentrations of manganese [20–23]. However, optical and SEM observations have revealed little appreciable dissolution of MnS inclusions.

5. Conclusions

Hot-forging at 900 or 1200 °C followed by intermediate holding at 800 or 1100 °C, respectively, prior to water quenching, resulted in a decrease in hardness and bend strength at holding times less than 50 s.

An abrupt strengthening occurred at an intermediate holding time of 50 s, though the prior austenite grain size gradually increased with intermediate-holding time. Hence there was little difference between the mechanical properties of the steels with no intentional holding time (2 s) and the holding time of 50 s. The strengthening phenomenon was more prominent as the forging temperature increased.

The concurrent appearance of microtwinning in the martensite and a simultaneous decrease in the activation energy values for tempering were seen without any significant change in the retained austenite content.

While direct evidence for carbon segregation has not been found, the strengthening phenomenon is explained by a carbon segregation model in which carbon segregates locally to defects introduced in austenite by hot-forging, followed by intermediate holding prior to quenching, thus resulting in local microtwinning and encouraging carbide precipitation during tempering.

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