Proposition of Two Parameters for a Good Characterisation of the Austenitising Condition of Microalloyed Steels

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Abstract. The characterisation of the compositional condition (particle dissolution/precipitation level), and the microstructural condition (austenite grain size and deformation state) are of vital importance for the subsequent phase transformations which occur during continuous cooling after thermomechanical processes of microalloyed steels. Thermoelectric power (TEP) measurement has been revealed as powerful method to analyse dissolution/precipitation processes together with recovery and recrystallisation phenomena. In this sense, this paper deals with characterising the austenitising condition by two fundamental parameters such as the prior austenite grain size (PAGS) and TEP. Likewise, to validate this hypothesis, the austenitising condition after a forging process in a medium carbon microalloyed steel has been simulated by a simple heat treatment which reproduce the same PAGS and TEP values. A comparison between the continuous cooling transformations occurred from both conditions, allow us to confirm that these parameters well characterise the austenite state of a microalloyed steel.

Introduction

During the hot–forge processes, steel experiences some microstructural changes caused by deformation, recrystallisation and grain growth processes, which can take place during each deformation and among the successive deformations. The resulting austenite microstructure will be decisive in the subsequent transformations which takes place in the steel during cooling, and produce the final microstructure that defines the mechanical properties wrought product.

Forge leads to grain size reduction by recrystallisation into ever finer grains. If the austenite is deformed without recrystallisation then the grains retain an elongated shape and contain additional defects such as dislocations which contribute to the energy stored in the austenite.

There is a role for the microalloying elements such as Ti, V and Al in thermomechanically processed steels. These serve to pin the austenite grain boundaries by forming carbonitrides and hence lead to further refinement. Therefore, to characterise the degree of dissolution/precipitation, austenite grain size and deformation state of austenite are of vital importance for the subsequent phase transformations which are carried out during continuous cooling after forging.

Thermoelectric power (TEP) measurement has been revealed as powerful method to study dissolution/precipitation processes together with recovery and recrystallisation

phenomena. In this sense, this paper deals with the analysis by a combination of TEP measurements and traditional metallographic techniques of the austenite state after forging, with the aim of characterising the austenite state by two fundamental parameters such as the prior austenite grain size (PAGS) and the TEP value. Likewise, to validate this hypothesis, it has been study the phase transformation carried out during continuous cooling in a medium carbon microalloyed with Ti and V steel.

Materials and Experimental Techniques

The experimental work has been done on a medium carbon, Ti-V microalloyed steel which its chemical composition is presented in Table 1.

Table 1.- Alloying elements in weight pct.

C	Mn	Si	S	Cr	Ni	Mo	V	Cu	Al	Ti	N
0.36	1.44	0.63	0.06	0.17	0.13	0.02	0.09	0.28	0.02	0.022	0.0096

The heat treatments were carried out using the heating and cooling devices of an Adamel Lhomargy DT1000 high-resolution dilatometer described elsewhere [1]. The heating device consists of a very low thermal inertia radiation furnace. The power radiated by two tungsten filament lamps is focussed on the specimen by means of a bi-elliptical reflector. The temperature is measured with a 0.1 mm diameter Chromel – Alumel (Type K) thermocouple welded to the specimen. Cooling is carried out by blowing a jet of helium gas directly onto the specimen surface. These devices ensure an excellent efficiency in controlling the temperature and holding time of isothermal treatments and as well as fast cooling in quenching processes.

Specimens were polished in the usual way for metallographic examination. Nital - 2pct etching solution was used to reveal the as-quenched microstructure by optical microscopy. The *PAGS* measurements were made on micrographs. The average grain size was calculated by using a linear intercept technique involving at least 50 intercepts, permitting the count of the number of grains intercepted by the grid line. The effects of a moderately non-equiaxed structure may be eliminated by counting the intersections of lines in four or more orientations covering all the observation fields with an approximately equal weight [2].

Thermoelectric power measurement is a powerful method that enables to study the amount of atoms in solid solution as well as the deformation state. The principle of the thermoelectric power measurement is the following: a sample is pressed between two reference blocks which are at different temperatures (ΔT). Due to the Seebeck effect, a potential difference ΔV is generated at the reference contacts. The relative TEP (ΔS) is then defined as the ratio $\Delta V/\Delta T$ with respect to the absolute TEP of reference blocks. For given temperature and microstructural state, it can be assumed that the different contributions to ΔS are additive. This assumption is valid as the material is low alloyed since the interactions between atoms can be neglected. Under this conditions, it can be written that the measured relative TEP signal of a sample is equal to the sum of different contributions:

$$\Delta S = \Delta S_{SS} + \Delta S_d + \Delta S_{pp} \quad (1)$$

Where ΔS_{SS} , ΔS_d and ΔS_{pp} are the contributions of solid solution, dislocations and precipitates, respectively. The elements in solid solution have a strong influence on the TEP values because they create new diffusion centres for electrons and phonons.

Concerning the effect of dislocations, it has been clearly established that a cold working, thus the introduction of dislocations, leads to negative TEP variation ΔS_d [3]. Finally, the main effect of precipitation on thermoelectric power is that it withdraws solute atoms from solid solution. However, the role of precipitates themselves is not completely understood. The coarse incoherent precipitates are thought to have no effect on the TEP, while the coherent precipitates can have influence on the TEP. These results can be explained by the fact that incoherent precipitates are similar to voids. During the formation of these precipitates, the measured TEP variations are thus essentially related to the decrease in solid solution content of the elements. In contrast, the coherent precipitates induce elastic and electronic disturbances in the lattice and thus modify the TEP.

Experimental an results

As mentioned in the previous section, TEP measurement is then a technique that enable to characterise the austenite deformation and precipitation state. Likewise, precise determination of austenite grain size is often essential in metallurgical studies of steel properties [4-7]. For example, fine grained size austenite usually transforms to fine ferrite, pearlite, bainite or martensite microstructures, thereby providing increase strength and toughness [8], *i.e.* the grain size is a microstructural factor which determine the austenite transformation kinetics during cooling. Therefore, it is concluded that the simultaneous measurement of TEP and PAGS can adequately characterise the austenite compositional and microstructural state.

Figure 1 schematically show the thermomechanical cycle of a usual industrial forge process. The forge process was simulated in a Hot Torsion Simulator. Samples were heated up to 1250 °C and held at this temperature by 15 minutes. Then they underwent a deformation of $\varepsilon=5$ with a deformation rate of $\dot{\varepsilon}=10s^{-1}$. Subsequently they were cooled at 4 °C/s to 1100 °C and then quenched.

Figure 1. Thermomechanical cycle to simulate industrial hot forge process.

Figure 2. Thermal cycle to reproduce the same TEP and PAGS conditions to those after forging.

With the aim of characterising the austenite state just before cooling (*i.e.* at 1100 °C), TEP and PAGS measurements were performed in as-quenched samples. The results obtained are shown in Table 2.

Table 2. TEP and PAGS measurements after thermomechanical cycle of Fig. 1

TEP, $\mu\text{V K}^{-1}$	PAGS μm
3.31 ± 0.2	52 ± 33

The same values of TEP and PAGS were able to be reproduced with a pure thermal cycle, where no deformation is included, such that schematically is presented in Fig. 2. Samples were heated at 10 °C/s up to 1250 °C, held at that temperature during 180 s, subsequently cooled at 4 °C/s to 1000 °C, maintained during 180 s and finally quenched. The PAGS and TEP obtained are given in Table 3. Since PAGS and TEP measurements after forging and after the thermal path are similar, it is assumed that the austenite compositional and microstructural states are also the same.

Table 3. TEP and PAGS measurements after thermal cycle of Fig. 2.

TEP $\mu\text{V K}^{-1}$	PAGS μm
3.43 ± 0.2	63 ± 15

In order to validate this hypothesis, samples after the thermomechanical treatment shown in Fig. 1 were subsequent cooled at the three different cooling paths listed in Table 4. The microstructures obtained (Fig. 3) consisted of allotriomorphic ferrite and pearlite (T1), martensite and bainite (T2) and acicular ferrite (T3).

Table 4. Cooling paths after austenitisation heat treatments

Cooling cycle	Temperatures	Cooling rate
T1	1000°C → 896 °C	5°C/s
	896 °C → 490 °C	1 °C/s
	496 °C → 300 °C	0.5 °C/s
T2	1000°C → 832 °C	17° C/s
	832°C → 600°C	10° C/s
	600 °C → 488 °C	6 °C/s
	488 °C → 300 °C	3 °C/s
T3	1000 °C → 850 °C	10 °C/s
	850 °C → 680 °C	5 °C/s
	680 °C → 560 °C	2°C/s
	560 °C → 300 °C	1°C/s

On the other hand, the same cooling cycles were performed on samples austenitised according to thermal cycle presented in Fig. 2. The microstructures obtained after cooling are presented in Fig. 4. It might be concluded from a comparison between Figs. 3 and 4 that the same austenite decomposition products are obtained, since the austenite state from which this microstructures are transformed is the same. Moreover, Vickers hardness measurements (Table 5) show similar values.

Figure 5. Microstructures obtained after the thermomechanical cycle presented in Fig. 1 at cooling path (a) T1, (b) T2, and (c) T3

Figure 6. Microstructures obtained after the thermal cycle presented in Fig. 2 at cooling path (a) T1, (b) T2, and (c) T3

Table 5. Vickers hardness measurements

	Thermomechanical processed samples	Thermal processed samples
T1	265±3	273±15
T2	463±36	427±26
T3	255±4	300±18

Conclusions

Austenitising conditions of a microalloyed steel after forging can be perfectly characterised by means of thermoelectric power and prior austenite grain size measurements. The same austenite state has been achieved with a thermal path with no deformation obtaining the same values for TEP and PAGS. This has been experimentally demonstrated because deformed and no deformed samples were cooled from 1100 °C with different cooling paths and the same microstructure was obtained. The measurement of the couple TEP-PAGS has shown to be an accurate technique to characterise the austenite compositional and microstructural state of a microalloyed steel.

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