JOURNAL OF MATERIALS SCIENCE 26 (1991) 5427–5432

Activation energy in the static recrystallization of austenite

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By means of torsion trials, the influence of various alloying elements on the parameter $t_{0.5}$ of Avrami's equation has been studied. It has been found that carbon accelerates the static recrystallization of the austenite and that the other elements, in solution or by forming precipitates, slow it down. It is verified that the activation energy is the parameter most sensitive to the chemical composition and its value has been defined in a collection of 11 steels of the C–Mn type and those, microalloyed with Nb and Ti. Finally an equation is proposed which allows the calculation of the activation energy as a function of the chemical composition of the steel with great accuracy.

1. Introduction

In recent years attempts have been made to make a mathematical model of the microstructural evolution of steels in the austenitic stage with the primary purpose of applying the model to shaping processes, especially to lamination.

The evolution of the austenitic grain when steel is subjected to one or several deformations is affected by a high number of variables, such as temperature, magnitude of the deformation, speed of the deformation, grain size, and chemical composition of the steel. It follows, therefore, that the construction of a mathematical model is not an easy task and only if a rigorous methodology is applied, will it be possible to construct a model which correctly interprets the successive variations in the austenitic grain. These variations are mainly due to the recrystallization which the austenitic grain undergoes during and after each deformation, that is to say, to dynamic and static recrystallization, respectively.

The recrystallized fraction of a static mode follows Avrami's law, which is expressed by

$$X_a = 1 - \exp\left[(-\ln 2)\left(\frac{t}{t_{0.5}}\right)^n\right] \quad (1)$$

where $t_{0.5}$ is the time corresponding to 50% of the recrystallized volume.

To know analytically the recrystallized fraction as a function of the elapsed time after deformation, it is, therefore, necessary to determine the value of the parameters $t_{0.5}$ and n. The influence of the above mentioned variables on the recrystallized fraction is shown mainly upon the parameter $t_{0.5}$ according to an equation whose expression, confirmed by various authors, is

$$t_{0.5} = A\varepsilon^p\dot{\varepsilon}^q D^s \exp\frac{Q}{RT} \quad (2)$$

where ε is the true deformation, $\dot{\varepsilon}$ the deformation speed, Q the activation energy, T the absolute temperature, $R = 8.318\ \mathrm{J\,mol^{-1}\,K^{-1}}$, D the grain size and p, q and s are parameters.

For some authors [1] the index n can be taken as a constant and for others [2] it depends also, although to a lesser degree, upon temperature and deformation.

The difficulty in establishing a valid model for all steels arises from the fact that the chemical composition of the steel influences notably the parameter $t_{0.5}$, each element of the alloy showing a specific influence. Nevertheless, in the literature [1, 2] we find that the activation energy (Q), or otherwise the parameter $t_{0.5}$, is not expressed as a function of the chemical composition of the steel and it only establishes a constant value for the C–Mn family of steels and another value for the steels microalloyed with Nb.

In this work we submit a study of the influence of C, Mn, Si, Mo, Nb and Ti on the kinetics of static recrystallization. The tests have been carried out in a torsion machine, and the recrystallized fraction has been determined by means of the double deformation method, selecting the so-called "back extrapolation" [3], and it has been proved that by using this method, the softened fraction and the recrystallized fraction are almost equal [4]. The activation energy has emerged as the most sensitive parameter to the chemical composition, depending to a greater or lesser degree upon all of the alloy forming elements. Our study, therefore, has concentrated upon determining Q in relation to the alloy elements mentioned above.

0022-2461/91 $03.00 + .12

TABLE I Chemical composition of steel (wt %) × 10^3

Steel	C	Mn	Si	Mo	Nb	Ti	N
1	622	546	159	–	–	–	–
2	849	727	258	–	–	–	–
3	191	826	0	–	–	–	–
4	180	1370	440	–	–	–	–
5	47	282	22	–	–	–	–
6	400	659	298	328	–	–	–
7	400	629	299	604	–	–	–
8	176	1266	279	–	–	22	6.6
9	174	1253	274	–	–	52	7.2
10	59	389	77	–	58	–	6.8
11	63	350	74	–	94	–	6

Although in other works [5] the influence of some alloy elements on the recrystallized fraction has been studied, the study was only qualitative.

2. Experimental procedure

The probes used in the tests had the following dimensions radius (r) = 3 mm, length of useful part (L) = 50 mm.

The deformation in the torsion trial is expressed by the number of turns and its equivalence with the true deformation is estimated according to

$$\varepsilon = \frac{2\pi r N}{\sqrt{3}L} \qquad (3)$$

The probes were heated by induction in a controlled atmosphere of argon and the heating temperature, prior to deformation, was always 1250 °C, with a maintenance time of 15 min.

A total of 11 steels whose compositions are shown in Table I were tested. They are steels with different compositions, including C–Mn type steels, C–Mo and microalloyed steels with Nb and Ti.

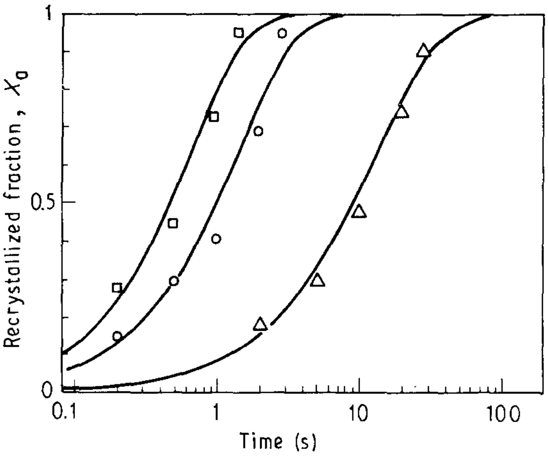

Figure 1 Recrystallized fraction plotted against time. Steel 4, $\varepsilon = 0.42$, $\dot{\varepsilon} = 3\ \mathrm{s}^{-1}$, $I = 450\ \mu\mathrm{m}$. (□ 1100 °C, ○ 1000 °C, △ 850 °C).

Finally, the test temperatures were always higher than Ar_3 and the deformation speed was $3\ \mathrm{s}^{-1}$.

3. Results of experiments

The curves which give the recrystallized fraction X_a were defined as a function of time, at different temperatures and with two deformations (0.21; 0.42). Fig. 1 shows an example of these curves, which correspond to steel 4. With the rest of the steels similar curves were obtained, that is to say, total recrystallization was achieved, even at temperatures close to Ar_3, except in the steels microalloyed with Nb in which, below a determined critical temperature, the recrystallization is only partial and consequently the 100% recrystallized fraction is not achieved, even though the times are long, in excess of 1000 s, as shown in Fig. 2 for steel 10.

All the curves which show total recrystallization follow Avrami's law, and according to Equation 2 a logarithmic representation of the parameter $t_{0.5}$ as a

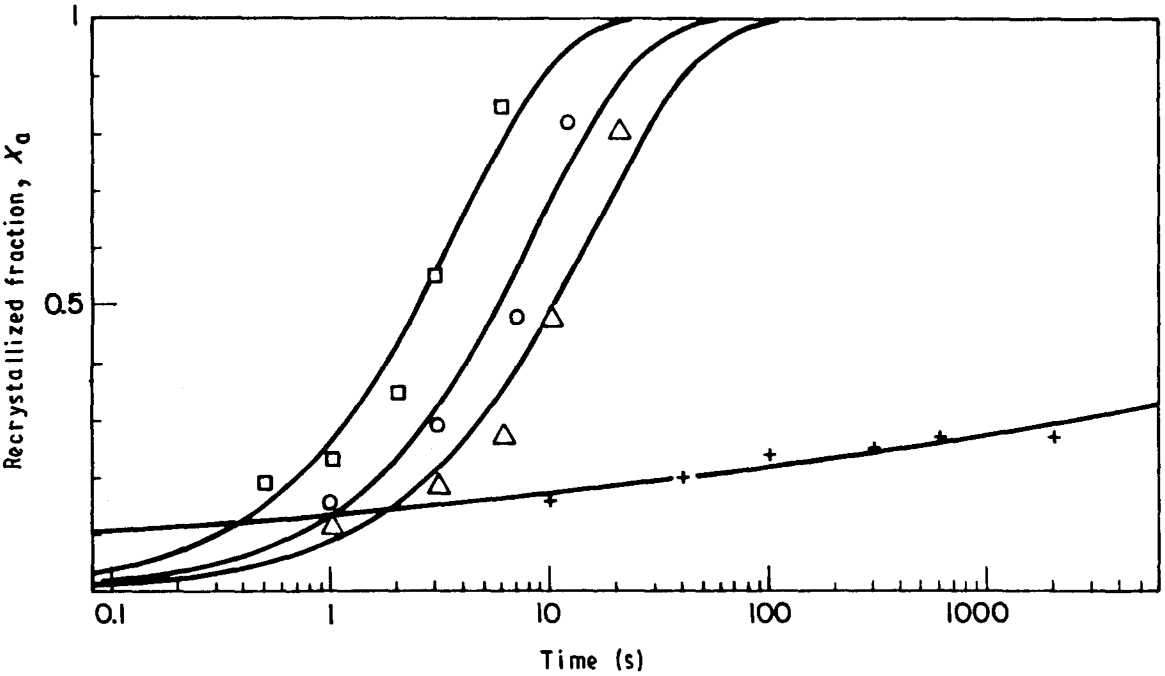

Figure 2 Recrystallized fraction plotted time. Steel 10, $\varepsilon = 0.21$, $\dot{\varepsilon} = 3\ \mathrm{s}^{-1}$, $I = 210\ \mu\mathrm{m}$. (□ 1150 °C, ○ 1100 °C, △ 1050 °C, + 1000 °C).

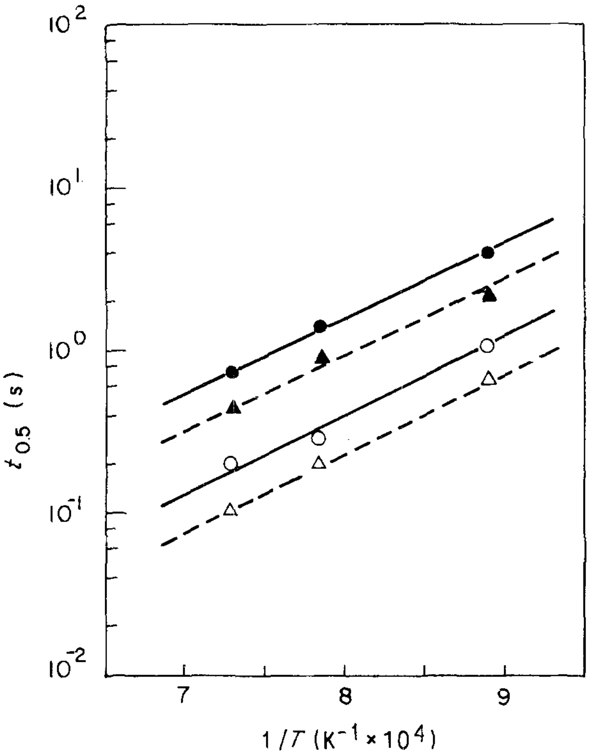

Figure 3 Parameter $t_{0.5}$ plotted against $1/T$. (● steel 1, $\varepsilon = 0.21$; ○ steel 1, $\varepsilon = 0.42$; ▲ steel 2, $\varepsilon = 0.21$; △ steel 2, $\varepsilon = 0.42$).

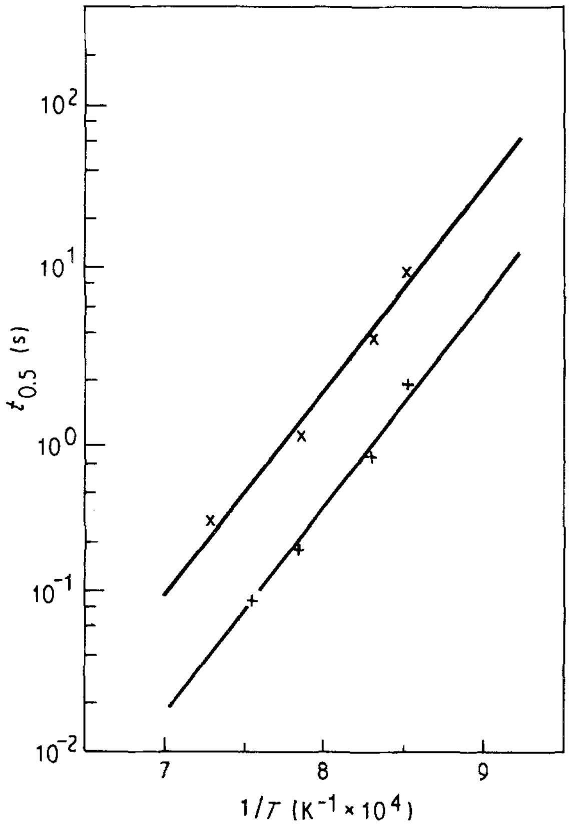

Figure 5 Parameter $t_{0.5}$ plotted against $1/T$. (× steel 5, $\varepsilon = 0.21$; + steel 5, $\varepsilon = 0.42$).

function of the reverse of the absolute temperature allows the determination of the value of Q.

The activation energy is independent of temperature and it can be considered roughly that it is also independent of deformation and grain size. Figs 3 to 8 shows the parameter $t_{0.5}$ plotted against $1/T$ for all the steels. The experimental values of Q and size of the initial grain (at 1250 °C, 15 min) for all the steels tested are shown in Table II. In the Nb steels, the activation energy is shown to be independent of the temperature

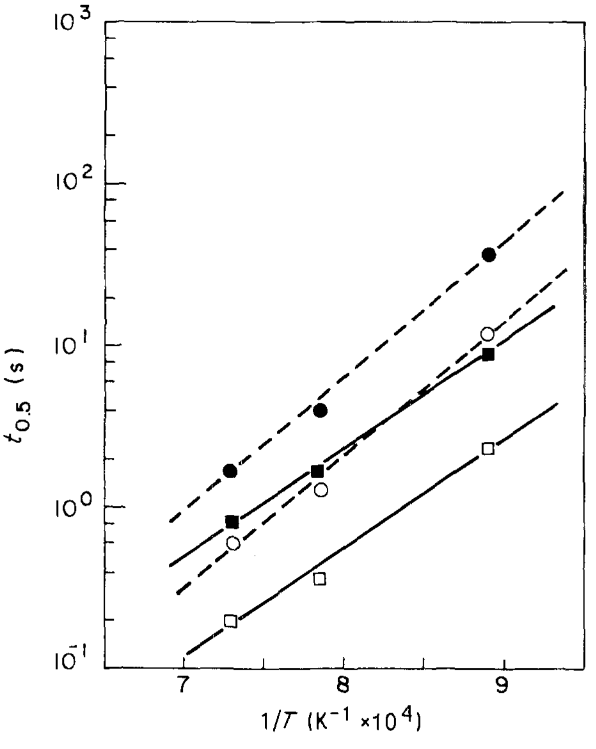

Figure 4 Parameter $t_{0.5}$ plotted against $1/T$. (■ steel 3, $\varepsilon = 0.21$; □ steel 3, $\varepsilon = 0.42$; ● steel 4, $\varepsilon = 0.21$; ○ steel 4, $\varepsilon = 0.42$).

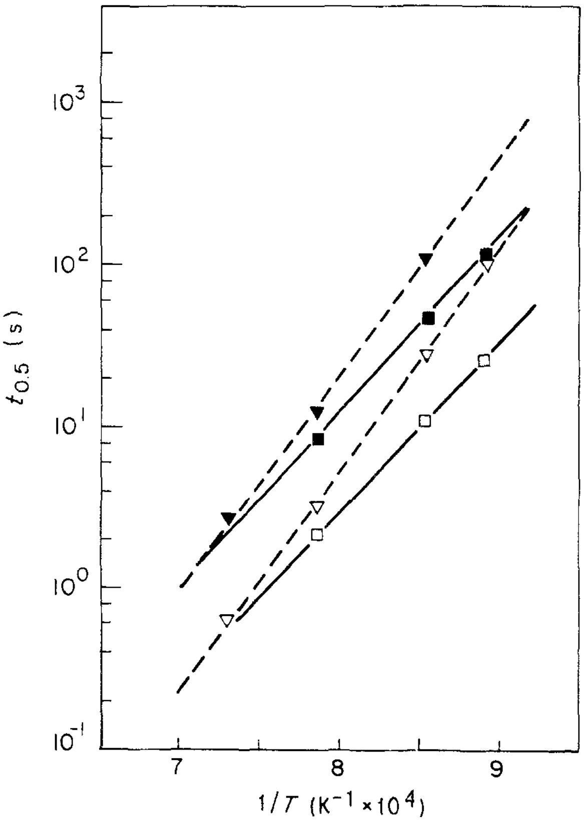

Figure 6 Parameter $t_{0.5}$ plotted against $1/T$. (■ steel 6, $\varepsilon = 0.21$; □ steel 6, $\varepsilon = 0.42$; ▲ steel 7, $\varepsilon = 0.21$; △ steel 7, $\varepsilon = 0.42$).

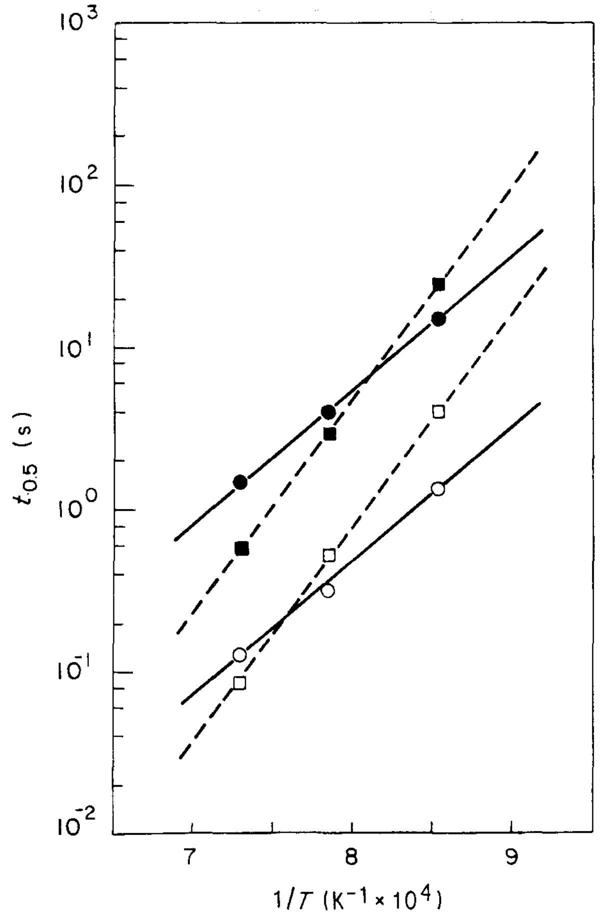

Figure 7 Parameter $t_{0.5}$ plotted against $1/T$. (● steel 8, $\varepsilon = 0.21$; ○ steel 8, $\varepsilon = 0.42$; ■ steel 9, $\varepsilon = 0.21$; □ steel 9, $\varepsilon = 0.42$).

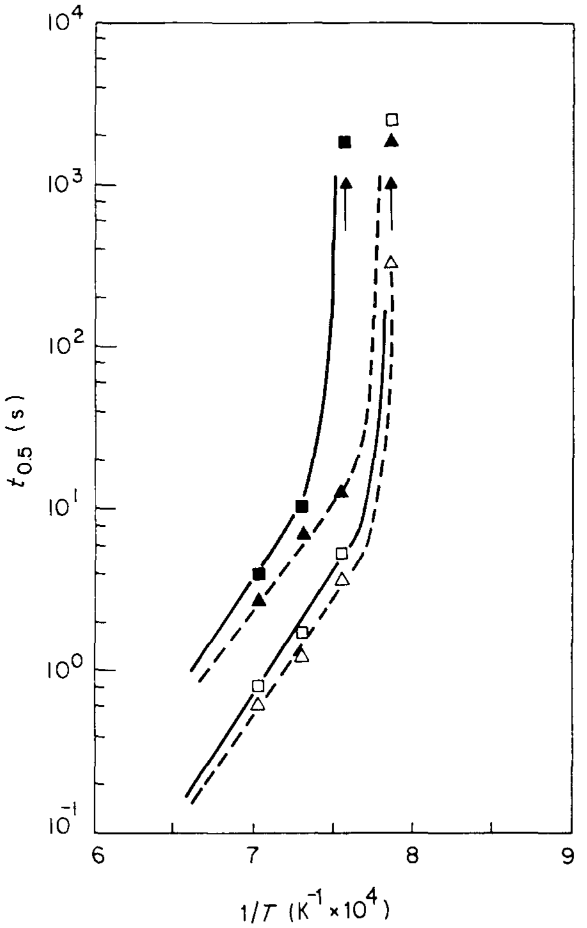

Figure 8 Parameter $t_{0.5}$ plotted against $1/T$. (▲ steel 10, $\varepsilon = 0.21$; △ steel 10, $\varepsilon = 0.42$; ■ steel 11, $\varepsilon = 0.21$; □ steel 11, $\varepsilon = 0.42$).

only up to the critical temperature, below which it starts to increase rapidly of temperatures between T_c and Ar_3, the activation energy undergoes a marked increase as can seen in Figs 9 and 10, which show the ln Q against $1/T$ in the steels 10 and 11, respectively. From these figures the value of the critical temperature is deduced demonstrating that it is a function of the Nb percentage and of the deformation (Table III).

TABLE II Experimental value of the activation energy (Q) and grain size (D)

Steel	Q (kJ mol^{-1})	D (μm)
1	92	325
2	92	165
3	130	270
4	159	450
5	243	235
6	205	310
7	260	340
8	159	75
9	251	80
10	276	210
11	293	195

TABLE III Critical temperatures (°C) in the steels microalloyed with Nb

Steel	ε	
	0.21	0.42
10	1050	1044
11	1100	1044

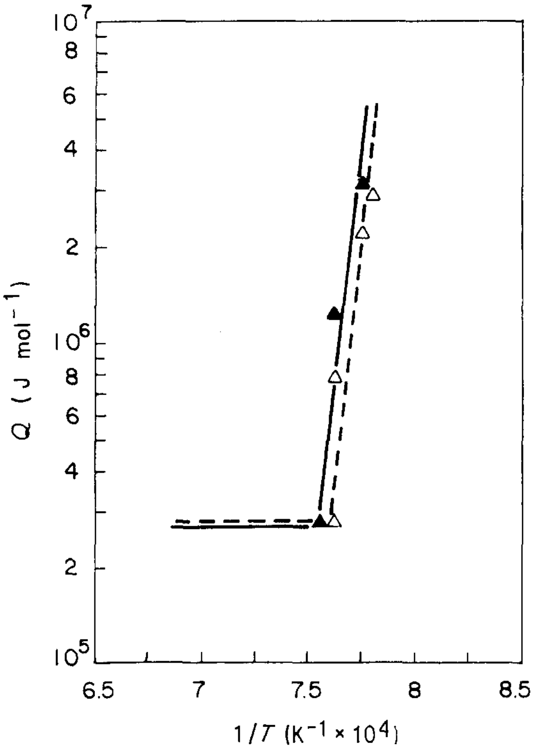

Figure 9 Activation energy plotted against $1/T$. (▲ steel 10, $\varepsilon = 0.21$; △ steel 10, $\varepsilon = 0.42$).

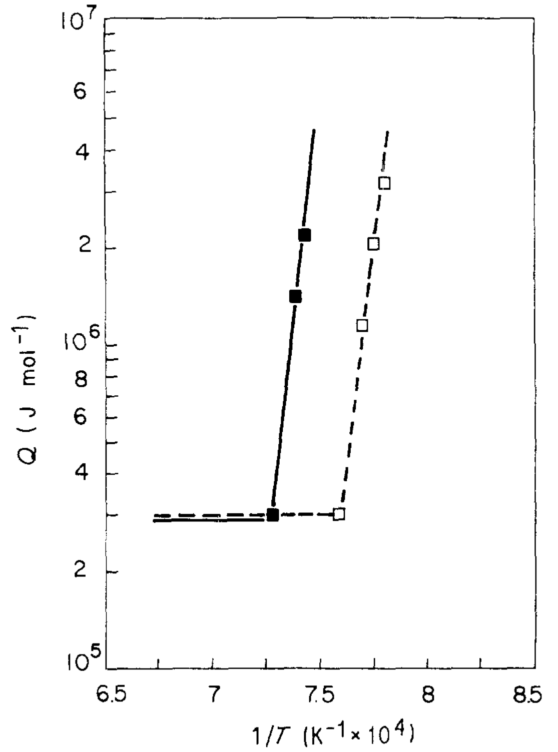

Figure 10 Activation energy plotted against $1/T$. (■ steel 11, $\varepsilon = 0.21$; □ steel 11, $\varepsilon = 0.42$).

4. Discussion of the results

In the group of C–Mn steels values of Q ranging from 92 000 J mol^{-1} for steels 1 and 2 to 243 000 J mol^{-1} for steel 5 have been obtained and this indicates, according to Equation 1, that the latter has a much slower recrystallization kinetics.

The C–Mo steels show relatively higher Q values, and amongst the microalloyed ones are the steels with Nb which present a higher activation energy and, therefore, a greater slowing of the recrystallization.

On the other hand, it is seen that an increase in the percentages of Mo, Ti and Nb result in an increase in Q. Similarly, an accurate analysis of the C–Mn steels allows one to deduce that an increase in the percentage of carbon originates an important decrease in the activation energy and an increase in the Mn and Si increases it.

If we take into account that the Mn and the Si are in substitutional solution in the austenite, that their atomic radii are somewhat larger than the atomic radius of the Fe and they are not precipitate forming elements, it can be estimated that their contribution to activation energy is the same at equal percentage. With the preceding hypothesis and making use of regression analysis, the following expression was defined for the activation energy

$$Q^* = 83\,600 + 978\,000 \frac{(\mathrm{Mn} + \mathrm{Si})^{0.537}}{\mathrm{C}^{1.269}} + 1728.7$$

$$(\mathrm{Mo})^{0.704} + 4952.10^{-5}(\mathrm{Ti})^{3.663} + 259.6(\mathrm{Nb})^{1.256} \tag{4}$$

the value of Q being given in J mol^{-1} and the contents of Mn, Si, etc. in wt % 10^3, exactly as expressed in Table I.

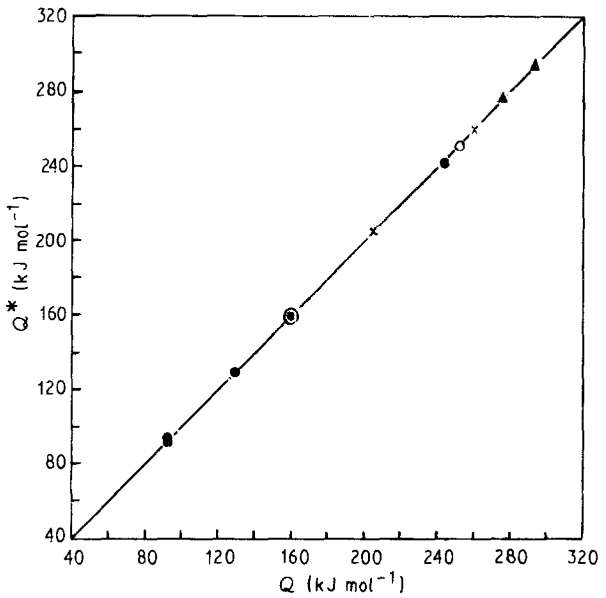

Figure 11 Calculated activation energy (Q^*) plotted against experimental activation energy (Q). (● C–Mn, × C–Mo, ○ C–Ti, ▲ C–Nb).

In Fig. 11 a graphic comparison between the experimental values of the activation energy (Table II) and those calculated according to Equation 4 is shown, in which we can observe an excellent degree of correlation between the two since all the points are situated on the bisecting line. Equation 4 is only applicable in the austenitic stage, that is to say is valid for temperatures higher than Ar_3, except for the steels with Nb in which the minimum temperature of application is the critical temperature. For the calculation of the temperature Ar_3 the following formula from the literature [4] can be used

$$Ar_3 = 902 - 527(\%\mathrm{C}) - 62(\%\mathrm{Mn}) + 60(\mathrm{Si}\%) \tag{5}$$

and which has been defined carrying out simulations in an experimental steel sheet mill.

On the other hand, it has been seen in the previous section that below the critical temperature the activation energy increases considerably and an analytical expression has been found which reflects quite accurately this behaviour. It consists of adding to the second member of Equation 4 the following term

$$Q' = A \exp\left(\frac{1\,100\,000}{RT}\right) \tag{6}$$

where

$$A = 8 \times 10^{-22}(\mathrm{Nb})^{-9.045} \cdot \varepsilon^{-3.189 \times 10^{-7}(\mathrm{Nb})^{3.687}}$$

The carbon content considered in Equation 4 is the total carbon, in interstitial solution in the austenite at the holding temperature (1250 °C), although a part of it has contributed during the cooling to the formation of the precipitates in the C–Mo steels and those microalloyed with Nb or Ti. Taking into account the more common stoichimetric relations in this type of precipitates, it follows that the amount of carbon present in them is very small as compared with the total carbon. On the other hand, the effect of the precipitates, whether they be carbides, nitrides or carbonitrides, on the activation energy depends on the percentages of the elements (Mo, Ti, Nb) which form them. Finally, the marked increase in the activation energy at temperatures below T_c in the steels microalloyed with Nb is obviously due to the blocking which the very fine Nb carbonitrides exert on the recrystallization. A simple estimation of the amount of Nb in solution, or precipitate, as a function of temperature, in accordance with the solubility equation [6, 7], would explain this behaviour.

4. Conclusions

This work leads to the following conclusions, amongst others:

1. The carbon in solution speeds up the recrystallization and the other elements slows it down.
2. In steel 8, microalloyed with titanium, the value obtained for the activation energy (159 000 J mol^{-1}) is due above all to the elements in solution and not to the precipitates (titanium nitrides), as can be shown using Equation 4.
3. The steels microalloyed with Nb exhibit the highest values of activation energy.
4. Equation 4 allows an estimation of the activation energy in the austenitic stage with a practically negligible error, except for the steels microalloyed with Nb in which it is only applicable at temperatures higher than the critical temperature. If Equation 4 was applied to the microalloyed steels containing Nb and Ti at the same time, it is still not known what the error would be and maybe it might be necessary to introduce a correcting factor.
5. The activation energy of the steels microalloyed with Nb is constant at temperatures higher than T_c and increases markedly as the temperature decreases below T_c. Equation 4 combined with Equation 6 allows an estimation of the activation energy in the range $Ar_3 \leqslant T \leqslant T_c$.
6. When the activation energy increases, the parameter $t_{0.5}$ also increases, as shows Equation 2, and therefore, Equation 4 will allow us to know in relative terms what the recrystallization kinetics of any two steels of the type studied will be for a given deformation and temperature.
7. The values obtained for the activation energy are valid when the deformation does not exceed the value ε_c, which marks the start of the dynamic recrystallization, since the deformations applied were less ($\varepsilon \leqslant \varepsilon_c$) for all the steels studied.

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Received 24 April 1989
and accepted 6 March 1991