Brittle-to-Ductile Transitions and its Relation to the Deformability of Cementite in Fully Pearlitic Steels.

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Abstract: Temperature dependence of absorbed impact energy in fully pearlitic steels was investigated. Two-step brittle-to-ductile transitions were observed. The value of the activation energy associated with the first transition is comparable to that of low carbon ferritic steels, indicating that the first transition is the brittle-to-ductile transition of ferrite phase in pearlitic steels. It suggests that the first transition is controlled by the dislocation glide in ferrite. The value of the activation energy associated with the second transition is higher than that of the first transition. Micrographs of fracture surfaces and side surfaces after fracture tests suggest that the activation energy associated with the second transition should relate to the deformability of cementite in pearlite, that is, the second transition is controlled by the dislocation activity in cementite of pearlite.

Keywords: BDT, Pearlite, Fracture, Cementite, Deformation

1. Introduction

Pearlitic steels have been widely used as structural materials. Possible parameters which control their mechanical properties are such as prior austenite grain size, block size, colony size, inter lamellar spacing and carbide thickness [1-3]. It is fairly in agreement that the inter-lamellar spacing controls the yield stress in the form:

$$\sigma_y = \sigma_o + kS_p^\alpha, \quad (1)$$

Where σ_y denotes the yield stress, σ_o and k are constants, S_p denotes inter-lamellar spacing where there is still an open issue whether $\alpha = -1$ or $-1/2$ [1, 2]. Fracture stress and deformability of pearlitic steels depend also on such parameters [2,4-11]. Salischvef *et al* [12] showed, using impact tests, that the reduction of the inter-lamellar spacing increases the value of energy for crack initiation and propagation in ferrite-perlite steels. TEM observation demonstrated that cleavage planes of fully pearlitic steels at low temperatures are $\{100\}$ planes of ferrite [13], and (001) , (120) , (100) or (001) in cementite [14]. Cuddy and Bassim [4] showed that a crack initiated at cementite due to the stress concentration near the ferrite-cementite interfaces propagate to ferrite in ductile fracture. Takahashi *et al* [15] concluded that block size is the controlling parameter of deformability of pearlitic steels, comparing with the relationships between the deformability and several parameters, which changed with heat treatment conditions.

A brittle-to-ductile transition (BDT), is one of the most important mechanical properties in steels, which is widely seen in bcc metals and covalent crystals. Fig. 1 shows a schematic drawing of the impact-absorbed energy as a function of the testing temperature in fully annealed ferritic steels with conventional grain sizes.

The absorbed energy, denoted by a solid line in Fig.1, keeps a lower level at low temperatures, which is called as a lower-shelf. Then, the absorbed energy significantly jumps around at a certain temperature. It is defined as the BDT temperature, T_{BDT} , by taking the mid-point temperature of the temperature range of the absorbed energy change. It is reported that BDT temperatures depend on the deformation rate. When the deformation rate in fracture tests increases, the BDT curves shift to the side of higher temperatures as shown with dotted line in Fig.1. St. John [16] demonstrated that the relationship between the deformation rate, $\dot{\epsilon}$, and the BDT temperature is given by the Arrhenius type equation:

$$\dot{\epsilon} = \epsilon_0 \exp\left(\frac{-G}{kT_{BDT}}\right), \quad (1)$$

where $\dot{\epsilon}$ is a strain rate, ϵ_0 is a constant, G is an activation energy of deformation process which controls the BDT, k is the Boltzmann constant, and T_{BDT} is the absolute BDT temperature. It has been elucidated that the value of the activation energy in Eq. (1) is close to that of the dislocation glide at around the BDT temperature, which indicates that the BDT is controlled by the thermally activated processes of dislocation glide [16-18]. The concept built in single crystalline materials has been also applied to the studies on the polycrystalline metals [19-21], suggesting that the controlling mechanism behind the BDT in multi-phase steels such as pearlitic steels can also be elucidated by experimentally determining the value of activation energy in Eq. (1).

Sirithanakorn *et al* reported [22] that there are two steps in the temperature dependence of absorbed energy in fully pearlitic steels, which denotes a two-step BDT in those steels. On the analogy of the fact that the onset of the BDT is controlled by dislocation glide in single phase materials with high Peierls potential as

mentioned, the two-step BDT should have different mechanisms behind the onset of each transition. In the present study, therefore, the impact-absorbed energy was firstly measured with different temperatures and deformation rates. The value of activation energy was obtained from the deformation rate dependence of the BDT temperature. The controlling process of the BDT in pearlitic steels was discussed, presuming that the dislocation glide is also the key mechanism behind the two-step BDT in fully pearlitic steels.

2. Experimental Procedures

SWRS92A patented fully pearlitic wires with the diameter of 5.5 mm were employed, the chemical composition of which is shown in Table1. Test pieces were firstly cut from the patented wires to be $1.5 \times 1.2 \times 20 \text{ mm}^3$ in dimension, and then three sides of the pieces were polished while a notched side was left as cut. The final dimension of the samples was approximately $1.2 \times 0.8 \times 20 \text{ mm}^3$. Fracture tests were carried with the impact blade speeds of 0.0033, 0.033, 0.33, and 3.3 m/s, respectively, followed by SEM observations using an angle selective backscattered electron detector in Ultra-55 (Carl Zeiss)

3. Results

Fig. 2(A) shows an SEM micrograph of the longitudinal section of the specimen after deep etching. The directions of cementite lamellae demonstrate the colony size is in the range of 5-15 μm showing that the grain orientations are randomly distributed. Fig. 2(B) shows an inversed pole figure map of the cross section. Fig. 2(C) is a grain map constructed from Fig. 2(B). Here each grain was filled

with one colour, where grain boundaries were defined with the miss orientation angle being larger than 15° . Black dots in the figure indicates the points where CI values are less than 0.1. It was found that the block sizes are in the range of 10 to 60 μm , with an average of 25.8 μm (by area).

Fig. 3 shows temperature dependences of absorbed energy obtained with the blade speeds of 0.0033, 0.033, 0.33, and 3.3 m/s, represented by solid symbols of black, red, blue and magenta, respectively. It was found that the absorbed energy is increasing with two jumps; the first jump appears around 10 kJm^{-2} at below 200K (20 kJm^{-2} at 228K in case of the 3.3m/s blade speed) and the second jump occurs around between 20 kJm^{-2} and 30 kJm^{-2} at above 200K (45 kJm^{-2} at 308K in case of the 3.3m/s blade speed). It suggests that there are twice brittle-to-ductile transitions in pearlitic steels, which is not seen in ferritic steels as illustrated in Fig. 1. Since the value of the absorbed energy is relating to dislocation activities, the two-step increase in the absorbed energy in pearlitic steels should be due to the different dislocation activities associating with each jump of the absorbed energy. Here, we defined the plateau of the absorbed energy at low temperatures below the first transition as a lower shelf and the second plateau of the absorbed energy at temperatures between the first and the second transitions as a middle-shelf, and the third plateau at temperatures above the second transition as an upper-shelf (take the slight decreasing region of the absorbed energy in case of the 3.33 m/s blade speed). Here, the first transition temperature was defined as the temperature that showed the middle value of the absorbed energy between the lower-shelf and middle-shelf. The second transition temperature was defined as the temperature that showed the middle value of the absorbed energy between the second-shelf and

the upper-shelf. The first transition temperatures with the blade speeds of 0.0033, 0.033, 0.33, and 3.3 m/s were 125K, 158K, 203K, and 228K, respectively. The second transition temperatures with the blade speeds of 0.0033, 0.033, 0.33, and 3.3 m/s were 268K, 273K, 316K, and 308K, respectively. Note, the data points near the transition temperature usually have significantly high values, especially in the case of the blade speeds of 0.0033 and 0.33 m/s, which might have some interesting relation with the BDT itself, however, the nature of the matter will be a subject of future studies. In order to elucidate the dislocation activities behind the two-step transition in the BDT of pearlitic steels, the activation energy were obtained from the deformation rate dependence of the transition temperatures of each step.

Fig. 4 shows Arrhenius plots from the first and the second transition temperatures as well as the BDT temperatures in the low carbon steel that exhibits a single transition. The tangent of the trend lines in Fig.4 gives the value of activation energy as shown in Eq. (1). The line with square symbols shows a result from the first transition temperatures, giving the activation energy of 0.13eV. The value of the activation energy is close to that of 0.16eV obtained from low carbon steels represented by circles symbols. Since the activation energy obtained from the deformation rate dependence of BDT temperatures is relating to that of dislocation glide, the agreement in the values of activation energies between the first transition in pearlitic steels and BDT temperatures in low carbon steel suggests that the first transition in pearlitic steels is controlled by the dislocation glide in ferrite phase of pearlitic steels. Fig. 5 exhibits the temperature dependence of the absorbed energy obtained from low carbon steels with the blade speed of 0.0033 m/s, where the result from pearlitic steel in Fig. 3 is superimposed. It shows the close values of BDT

temperatures between the pearlitic steels of 125K and the low carbon steel of 113K although the absolute values of the absorbed energy are different. This agreement of BDT temperature also supports the idea that the first transition occurs by increasing ductility of the ferrite phase. The value of the active energy from the second transition shown in Fig. 4 was 0.78 eV, which is significantly higher than that obtained from the first transition. This means that the controlling process of the second transition is not dislocation glide in the ferrite phase. The controlling mechanism behind the first and second transitions will be discussed in the next section. It is worth pointing here that the extrapolation lines from the first and second transitions in Fig. 4 appear to intersect each other at the blade speed of two orders of magnitude higher, which means that the first and the second transition temperature in Fig. 4 exchange each other at a particular blade speed.

4. Discussion

The similarity of activation energies between those obtained from the first transition temperature and BDT temperatures of low carbon ferritic steels suggests that the lower transition is relating to the dislocation glide in ferrite phase. Fig. 6 shows SEM images of fracture surfaces from the specimen fractured at the lower-shelf (173K), the middle-shelf (233K), and the upper-shelf (343K), respectively. The blade speed for the impact tests was 0.33 m/s. There is a very distinctive difference in morphology of fracture surfaces between the specimens fractured in the lower/middle-shelf and that fractured in the upper-shelf. Figs. 6(A) and (D) show that the fracture unit size is in the range of 20-50 μm is close to the block size shown in Fig. 2(A). Enlarged images in Figs. 6(B), (C), (E), and (F) show river patterns,

suggesting the fracture surfaces are {100} cleavage planes of ferrite. Figs. 6(H) and (I) show the enlarged fracture surfaces of Fig. 6(G) from the specimen fractured in the upper-shelf, indicating ductile fracture although they are not typical ductile fracture surfaces such as dimple patterns. The size of small deformation units observed in Fig. 6(I) is about 500 nm that is about 3-5 inter-lamellar spacing, suggesting a very different deformation process compare to a conventional ductile fracture process that results in dimple patterns. Since Fig. 6 suggests that the second transition is relating to the plastic deformation of cementite, each side surface of a sample taken near the notch was also observed.

Fig. 7(A), (C), and (E) show side surfaces near the crack in the specimens fractured at the lower-shelf, the middle-shelf, and the upper-shelf respectively. Enlarged images in Fig. 7(B) nor (D) do not indicate the obvious signs of plastic deformation but micro-cracks extending on the side surfaces. The cracks penetrate cementite lamellae, indicating that cementite lamellae are brittle at the temperatures in both the lower- and middle-shelves. Fig. 7(E) indicates that any micro-crack was not observed on the side surface of the sample fractured in the upper-shelf. Fig. 7(F) shows an enlarged image in Fig. 7(E) demonstrating that micro deformation bands that are denoted by arrows are passing through the cementite lamellae. Some micro-deformation bands were able to pass into adjacent colonies. The second transition in Fig. 3 must be relating to the plastic deformation in cementite. This is derived from the facts that the absence of micro-cracks penetrating cementite lamellae in the specimens fractured both in the lower- and middle-shelves, the temperatures of which are lower than the second transition temperature. Together with the deformation bands are observed in the specimen

fractured in the upper-shelf, the temperature of which is higher than the second transition temperature. It is to be emphasized that the deformation of cementite plays important role on the BDT in fully patented pearlitic steels.

The number of studies on the deformation of cementite is quite limited since it has been difficult to produce single-phase cementite large enough in dimension for having mechanical tests. The compression tests of bulk cementite samples produced by using sintering methods elucidated that the cementite deforms plastically and superplastically at above 573K and 873K, respectively. Bending tests indicated that the BDT temperature is between 873K-923K under crosshead speed of 0.5 mm/min [23]. The second transition temperature in the pearlitic steel used in this study was between 268K and 308K as shown in Figure 3, which is much lower than that in the BDT temperature in single phase cementite. It may be due to the difference in the deformability of cementite being as a single phase or as lamellae structure in pearlite. Although the single-phase cementite is hard to plastically deform around room temperature, cementite in pearlite is capable of plastic deformation around room temperature, Terashima *et al* [23] showed, using TEM, abundant dislocations in cementite of pearlite after cold rolling at room temperature. Tanaka *et al* [24] also demonstrated that cementite in pearlitic steels can plastically deform even in tensile tests at room temperature, measuring the value of plastic strain with precision markers. Ohasi *et al* [25] pointed, using finite element method, that the local deformation of cementite is prohibited by the uniform deformation of adjacent ferrite in pearlite, which enables the cementite in pearlite to deform uniformly without forming shear bands. The characteristic property of cementite is expected by the lamellar structure of ferrite and cementite in pearlite.

Another TEM observation reported [14] the effect of deformation temperature on dislocation density in both ferrite and cementite phases in pearlite. It was elucidated that the dislocation density in ferrite is approximately one order higher than that in cementite after tensile tests below 500 K. The TEM observation also elucidated a large number of dislocations in ferrite near the interface between ferrite and cementite and few numbers of dislocations in cementite after a tensile test at room temperature, which indicates that dislocations in ferrite are firstly activated and followed by those in cementite. This also supports that the first jump of the absorbed energy in the pearlitic steels as seen in Fig. 3 is controlled by the dislocation activity in ferrite, and that the second jump is controlled by the dislocation activity in cementite. Hence, the large value of activation energy of 0.78 eV obtained from the second transition is presumed to be the activation energy of dislocation glide in cementite in fully pearlitic steels.

5. Conclusion

The brittle-to-ductile transition in fully pearlitic steels was investigated. The temperature dependence of the absorbed impact energy was measured with the blade speed of 0.0033, 0.033, 0.33 and 3.3 m/s. The absorbed energy per unit area increased with two steps as the test temperature increased: the first jump of around 10 kJm^{-2} (25 kJm^{-2} in the case of 3.3 m/s blade speed) in the temperature between 158K and 228K, and the second jump of around 22 kJm^{-2} (45 kJm^{-2} in the case of 3.3 m/s blade speed) in the temperature between 268K and 316K. The value of activation energy associated to the first transition was measured to be 0.16eV, which is very close to 0.13eV of the low carbon ferritic steels. The energy associated

to the second transition was measured to be 0.78eV. The twice jumps of the absorbed energy suggests a two-step brittle-to-ductile transition in fully pearlitic steels. SEM images of side surfaces exhibited that micro-cracks propagated through cementite lamellae at temperatures below only those of the second jump of the absorbed energy, suggesting that the deformability of cementite controls the second jump.

Acknowledgment

The authors appreciate that the part of this work was supported by Japan Science and Technology Agency (JST) under Collaborative Research Based on Industrial Demand “Heterogeneous Structure Control: Towards Innovative Development of Metallic Structural Materials”.

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Fig. 1 Schematic drawing of impact-absorbed energy as a function of temperature with 2 different strain rates.

Table 1 Chemical compositions of SWRS92A used.

C	Si	Mn	P	S	Cu
0.90-0.95	0.12-0.32	0.60-0.90	<0.025	<0.025	<0.20

Fig. 2 (A) is SEM micrograph of the cross section of the wire after deep etching, (B) and (C) are inverse pole figure map of ND and the unique colour grain map constructed with the criteria of 15° difference, respectively. (cf. online version for coloured image)

Fig. 3 Temperature and blade speed dependences of the absorbed energy per unit area.

Fig. 4 Arrhenius plots from the first and the second transition temperatures in pearlitic steels. An

Arrhenius plot of BDT temperature in low carbon ferritic steel is also shown in the figure.

Figure 5 Comparison of temperature dependence of absorbed energy per unit area between of pearlitic steels and low-carbon ferritic steels near the first transition temperature of the ferritic steel at blade speed of 0.0033 m/s.

Fig. 6 (A), (D), and (G) show typical fracture surfaces fractured in the lower shelf, middle shelf and upper shelf, respectively. (B) and (G) are enlarged image in (A). (E) and (F) are enlarged image in (D).

(H) and (I) are enlarged image in (G).

Fig 7 SEM images of side surfaces near fracture surfaces. (A), (C), and (E) are from the specimen fractured in the lower-shelf, middle-shelf and upper-shelf, respectively. (B), (D), and (F) are enlarged images from the rectangles in (A), (C), and (E), respectively.