Lattice Defects Revealed by Hydrogen Thermal Desorption Analysis of Carbon Steel Strained with/without Hydrogen Pre-charging

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Effects of cyclic straining on the development of lattice defects were studied in a medium carbon steel containing globular cementite, using thermal desorption analysis (TDA) of post-charged hydrogen that acts as a tracer. The TDA curves were statistically analyzed using Gaussian function in order to separate comprised sub-peaks. The influence of hydrogen pre-charging was further explored. TDA desorption curves were separated into dislocations, grain boundaries, vacancies and vacancy clusters assuming Gaussian distributions for making quantitative comparisons of each defect. Tensile straining of 0.04 readily forms small amount of vacancies. Cyclic straining of 0.004 strain amplitude was more effective in vacancy formation, followed by their clustering. Hydrogen pre-charging before strain cycling was effective in enhancing the formation of those kinds of lattice defects. The maximum fractions of peak areas due to vacancies and vacancy clusters attained in the present study are 21.9% and 6.5% of the total desorption respectively. It was also revealed that the amount of hydrogen in the desorption peak from dislocations increases by straining in the presence of hydrogen. Slow tensile straining was more notable than cyclic straining, while the latter was increased by intensified hydrogen pre-charging.

KEY WORDS: tensile straining; cyclic straining; thermal desorption analysis; hydrogen trap; hydrogen pre-charge; Gaussian distribution; vacancy; vacancy cluster; dislocation; grain boundary.

1. Introduction

Under circumstances where hydrogen enters, steels are degraded in ductility or strength, leading to immature fracture. This phenomenon is called hydrogen embrittlement and should be avoided in the design or operation of plants or machines.

Almost all hydrogen in steels is trapped in nanoscopic open volume like the lattice defects; dislocations, vacancies, grain boundaries, and precipitate/matrix interface. When hydrogen is trapped by these lattice defects, it alters their nature. This in return affects the deformation behavior or fracture, and causes the degradation in ductility or strength. The state of hydrogen in steel is important for studying hydrogen embrittlement.

When a steel is strained, lattice defects are additionally produced and finally they lead the steel to failure. In hydrogen embrittlement, this process may be enhanced by the entry of hydrogen. Straining mode, monotonic or cyclic straining, substantially affects embrittlement associated with respective behaviors of various lattice defects. It is also important to examine the development of lattice defects by straining with/without hydrogen pre-charging.

Increase of hydrogen pick-up by straining is reported in pure iron, low carbon ferritic steels and Inconel 625, and it is discussed to result from the formation of vacancies and their clusters.\(^1\)\(^-\)\(^3\) It is also reported that straining after hydrogen pre-charging results in the creation of increased amount of point defects, presumably vacancies.\(^3\) In the case of high cycle fatigue loading for martensitic steel, vacancy formation is concluded through the change of hydrogen pick-up.\(^4\) Unfortunately peak separation in TDA curves or quantification of them has not been achieved. There are very few reports on the results of slow strain rate tests, especially strain controlled cyclic straining, where strain rate is one of the basic factors that determine the deformation or fracture.

Thermal desorption analysis\(^5\)\(^,\)\(^6\) is a powerful tool for revealing hydrogen traps. The desorption curve is basically described by McNabb-Foster equation\(^7\) and some approximate solutions have been proposed\(^8\)\(^-\)\(^9\) because of the difficulty of general solution. Numerical simulation is presented in case of dissociation-controlled or diffusion-controlled hydrogen desorption. However, because of inevitable assumptions for parameters, precise analysis is difficult.

When the TDA curve is not a single peak, it implies involvements of thermal dissociation-controlled desorption. Simulation methods are not settled for the case where various types of trap sites are included. Gaussian functions for sub-peaks and their superimpositions have been often
assumed for describing a TDA curve.\textsuperscript{10,11)}  

In a previous report,\textsuperscript{12)} a statistical analysis by Gaussian was applied to the TDA desorption curves obtained from low and medium carbon steels that had been given normalizing heat treatment. The TDA curves were tentatively separated into sub-peaks, and they were correlated with dislocation and grain boundary hydrogen traps. Then the method was verified by applying to the desorption curves from specimen with different amount of impurity phosphorus.\textsuperscript{12)}  

It was further verified by applying to a set of desorption curves that were hydrogen charged at different current densities.\textsuperscript{12)}

This paper treats a medium carbon steel used in Ref. 12), but with globular cementite. After monotonic tensile straining, hydrogen post charging was conducted. This post charged hydrogen acts as a tracer for examining the behavior of lattice defects by TDA. The measured desorption curves were analyzed by Gaussian function to separate sub-peaks. The newly produced sub-peaks, which were different from above-mentioned dislocation and grain boundary peaks, were correlated with the lattice defects created by straining. The effect of cyclic straining was also examined. The effect of hydrogen pre-charging before straining was further explored. Each sub-peak was discussed with respect to the effects of straining and hydrogen pre-charging on the formation of lattice defects quantitatively.

\section{Experimental Procedure}

A commercial JIS S45C was used for the experiments, whose chemical composition (check analysis) is summarized in Table 1. It was as-rolled bar stock used in the previous report,\textsuperscript{12)} except a spheroidizing heat treatment by slow cooling from 1 033 K to 873 K at rate of 10 K/h. Lamellar cementite in pearlite was changed to globular shape.

The round bar specimens whose parallel portion was 13 mm in diameter and 20 mm in length were machined from the center of the heat treated bars. These specimens were subjected to one of the following straining. Namely, i) no straining, ii) 0.04 tensile straining at slow strain rate of 10\textsuperscript{–3}/s, and iii) 100 cycles of push-pull straining with total strain amplitude of 0.004 at strain rate of 10\textsuperscript{–3}/s. Servo hydraulic fatigue testing machine was used for straining; straining was measured and controlled by strain gauge of the testing machine, followed by fixing the strain gauge. The time span of this manipulation was less than 7 minutes, before the specimen surface returns almost to room temperature.

Final TDA measurement was conducted within 1 hour storage in liquid nitrogen. Hydrogen was detected by gas chromatograph with the heating rate of 100 K/h.

Table 2 summarizes the combination of all test conditions.

\begin{center}
\begin{table}[h]
\centering
\begin{tabular}{|c|c|c|c|c|c|}
\hline
Symbol & H. pre-charge & Straining & H. post charge \\
\hline
#0 & none & None & X3 \\
#4\% & ditto & 4×10\textsuperscript{–2} tensile strain & ditto \\
H4\% & X3 & ditto & ditto \\
#100 & none & $\pm4\times10^{-3}$, 100 cycles & ditto \\
H100 & X3 & ditto & ditto \\
BH100 & Y6 & ditto & ditto \\
\hline
\end{tabular}
\caption{Summary of conditions for hydrogen pre-charge, straining and hydrogen post charge.}
\end{table}
\end{center}

3. Results

\subsection{Desorption Curve Profile Analysis}

The hydrogen desorption curve of specimen #0, which has not been subjected to straining, is shown in Fig. 1. Although the specimen has fan-shaped cross-section, the desorption curve has essentially the same feature with that of round bar specimen reported in the previous paper.\textsuperscript{12)} It has maximum around 330 K and has a shoulder at 365 K.

This desorption curve has been separated into 2 sub-peaks of Gaussian functions. Fitting of each peak was conducted using Gaussian functions of the following form for the convenience.

\[ D(T) = PH \times \exp \left( -\frac{(T - PT)^2}{2 \times PW^2} \right) \]

Here \( D(T) \) is desorption rate at temperature \( T(K) \), fitting parameters \( PH, PT \) and \( PW \) are peak height (mass ppm/min), peak temperature (K) and peak width (K). The \( PW \) is related to the effects of straining and hydrogen pre-charging on the formation of lattice defects quantitatively.

\begin{center}
\begin{table}[h]
\centering
\begin{tabular}{|c|c|c|c|}
\hline
Steel & C & Si & Mn & P & S & Cu & Ni & Cr & Mo & Al & N \\
\hline
45B & 0.46 & 0.21 & 0.81 & 0.019 & 0.019 & 0.03 & 0.01 & 0.14 & <0.01 & 0.033 & 0.0055 \\
\hline
\end{tabular}
\caption{Chemical composition of SCM435 steel.}
\end{table}
\end{center}
to the standard deviation, and the full width at half maximum (FWHM) is expressed in the following form.

$$FWHM = \sqrt{2ln(2)} \times PW$$  \hspace{1cm} (2)

The amount of desorption $H$ (mass ppm) of each sub-peak can be calculated by

$$H = PH \times PW \times \sqrt{2\pi \times 60} / HR$$  \hspace{1cm} (3)

where $HR$ is the heating rate (K/h).

As shown in Fig. 1, TDA curves are decomposed to lower (A1) and higher (A2) temperature peaks which were ascribed to dislocations and grain boundaries.\(^{12}\) Fitting parameters, peak height ($PH$), peak temperature ($PT$) and peak width ($PW$) are summarized in Table 3. Symbols “#” and “H” in Table 2 are for specimens without or with hydrogen pre-charging, respectively. Continuing two or three figures mean the condition of straining, “0” for no straining, “4%” for tensile straining by 0.004 (4%), and “100” for straining 100 cycles.

3.2. Slow Strain Rate Tensile Straining

Figure 2 illustrates the stress-strain relationship of the slow strain rate tensile strain up to strain of 0.04. Tensile stress tends to increase by hydrogen pre-charging. Two specimens with and without hydrogen pre-charging, were subjected to TDA measurement after hydrogen post-charging. The results are shown in Figs. 3(a) and 3(b).

Figure 3(a) is the results of specimen #4% strained up to 0.04 without hydrogen pre-charging. Comparing with Fig. 1, peak height around 330 K is higher and it also has shoulder around 365 K. Peak separation by Gaussian function has been applied using the same peak temperature and peak width with specimen #0. It is almost fitted by the synthesis of peaks A1 and A2 but small desorption remains around 385 K. It is the hydrogen desorption from a newly produced lattice defect. This third peak B is fitted by Gaussian function as shown in Fig. 3(a). All the fitting parameters of #4% are also summarized in Table 3. The parameters of peak B are less reliable because of the weakness of the residue.

Figure 3(b) is the measured desorption curve of specimen H4%, which has received both pre-charging and tensile straining. The maximum desorption is almost same with specimen #4% in Fig. 3(a) but the desorption curve extends to higher temperature side. This desorption curve is fitted by A1 and A2 peaks using the peak temperature and peak width of #0 in Table 3, as the case of Fig. 3(a). There remains more desorption beyond 370 K. Referring to Fig. 3(a), peak B is subtracted from the residue, using the peak temperature of specimen #4%. Even if the peak temperature of the peak B is set to 393 K, which is higher than 385 K of #4%, there still remains desorption unexplained by one Gaussian function. This residue is named as peak C and fitted by a new Gaussian function. Measured desorption curve can be separated into 4 Gaussian peaks. These peaks and the synthesis

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### Table 3. Summary of desorption curve fitting for not pre-charged nor strained (#0%), pre-strained 4% (#4%) and pre-charged and strained 4% (H4%). All specimens are post charged by X3 condition before TDA measurements.

| Symbol | Item       | A1      | A2      | B       | C       |
|--------|------------|---------|---------|---------|---------|
|        | Height, ppm/min | 0.01    | 0.0037  | --      | --      |
| #0%    | Temp., K    | 333     | 368     | --      | --      |
|        | Width (SD) K| 20      | 12      | --      | --      |
|        | Peak height ratio (A2/A1) | 0.37    |          |         |         |
|        | Height, ppm/min | 0.027   | 0.0095  | 0.001   | --      |
| #4%    | Temp., K    | 333     | 368     | 385     | --      |
|        | Width (SD) K| 20      | 12      | 10      | --      |
|        | Peak height ratio (A2/A1) | 0.35    |          |         |         |
| H4%    | Temp., K    | 333     | 368     | 393     | -410    |
|        | Width (SD) K| 20      | 12      | 11      | -12     |
|        | Peak height ratio (A2/A1) | 0.58    |          |         |         |

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Fig. 1. Results of curve analysis for not pre-charged nor strained (#0%). Post hydrogen charge condition is X3.

Fig. 2. Comparison of stress-strain curves during slow strain rate tensile tests; strained 4% without pre-charging (#4%) and after pre-charging with X3 condition (H4%). All specimens are post charged with X3 condition before TDA measurements.
of them are shown in Fig. 3(b). All the fitting parameters of H4% are also summarized in Table 3.

Figure 4 compares the peak heights of specimens #0, #4% and H4%. Slow tensile straining without hydrogen pre-charge yields peak B, in addition to the increased heights of peaks A1 and A2. Hydrogen pre-charge before tensile straining not only makes peak B higher but further creates peak C. The height of peak A1, which means the amount of hydrogen trapped by dislocations, depends on the degree of pre-straining but not on hydrogen pre-charging. On the other hand, peak A2, i.e. grain boundary traps, depends not only pre-straining but also on hydrogen pre-charging clearly.

3.3. Cyclic Straining

Figure 5 illustrates the hysteresis curves of 100th cycles for hydrogen not pre-charged specimen. Hydrogen pre-charged one exhibits almost same hysteresis curve as Fig. 5. Although the total strain amplitude is 0.004, the specimen receives almost 0.006 inelastic strain including Bauschinger strain, during both tensile and compression cycles.

Figure 6(a) shows the TDS desorption curve of the specimen #100, which was not subjected to hydrogen pre-charge but to 100 straining cycles. The maximum desorption rate takes place around 330 K just same with #4% in Fig. 3(a), but it outskirts beyond 425 K.

This desorption curve has also been analyzed in terms of Gaussian function. The procedure is basically same with #4% of Fig. 3(a). First, desorption curves from dislocation (A1) and grain boundary traps (A2) are fitted using the peak temperatures and peak widths in Table 4; they are same with #0, #4% and H4% in Table 3. The residue is further fitted by Gaussian functions using the same parameter as H4% in Table 3, creating peak B and C. The synthesis of peaks A1, A2, B and C is in good agreement with the measurement. All the fitting parameters of #100 are summarized in Table 4.
Figure 6(b) shows the results of measurement and fitting by Gaussian functions of specimen H100, which has been subjected to hydrogen pre-charge before cyclic straining. The fitting has been executed just same with #100 in Fig. 6(a). The peak C widely spreads and requires higher peak temperature and larger peak width for the improved fitting. All the fitting parameters are summarized also in Table 4.

Figure 7 compares #100 and H100 with #0 in the peak height of each sub-peak. Figure 7 is similar to Fig. 4 for the case of monotonic tensile straining. A big difference between the two figures exists in the height of peak C. It becomes higher in case of cyclic straining irrespective of hydrogen pre-charging.

### Table 4. Summary of desorption curve fitting for strained 100 cycles without hydrogen pre-charge (#100) and strained 100 cycles after hydrogen pre-charging (H100). Both specimens are post charged before TDA measurements. Hydrogen charging was conducted with X3 condition.

| Name | Item      | A1  | A2  | B   | C   |
|------|-----------|-----|-----|-----|-----|
| #100 | Height, ppm/min | 0.0305 | 0.0108 | 0.0036 | 0.0009 |
|      | Temp, K   | 333 | 368 | 393 | 410 |
|      | Width, K  | 20  | 12  | 11  | 12  |
|      | Peak height ratio (A2/A1) = 0.35 |
| H100 | Height, ppm/min | 0.0298 | 0.0110 | 0.0080 | 0.0048 |
|      | Temp, K   | 333 | 368 | 393 | 418 |
|      | Width, K  | 20  | 12  | 11  | 16  |
|      | Peak height ratio (A2/A1) = 0.37 |

Figure 6(b) shows the results of measurement and fitting by Gaussian functions of specimen H100, which has been subjected to hydrogen pre-charge before cyclic straining. The fitting has been executed just same with #100 in Fig. 6(a). The peak C widely spreads and requires higher peak temperature and larger peak width for the improved fitting. All the fitting parameters are summarized also in Table 4.

Figure 7 compares #100 and H100 with #0 in the peak height of each sub-peak. Figure 7 is similar to Fig. 4 for the case of monotonic tensile straining. A big difference between the two figures exists in the height of peak C. It becomes higher in case of cyclic straining irrespective of hydrogen pre-charging.

### 3.4. Intensified Hydrogen Pre-charge

Hydrogen pre-charging was changed from X3 condition to Y6 conditions in order to increase the hydrogen pick-up. The amount of poison NH₄SCN and current density are described in bottom of Table 2. Straining and post-charging conditions were same with H100. Result of measurement is
Hydrogen exists basically at the interstitial sites in metals but will be more stable with larger nanoscopic open volume. As mentioned in section 1, vacancy formation by straining is reported in various materials. In a cold drawn pure iron, formations of vacancies and vacancy clusters have been confirmed by TDA measurement in combination with observation of sub-structure during recovery and recrystallization. Straining of a low-carbon ferritic steel caused increase of hydrogen pick-up and the increase disappeared by heating the strained specimen at above 473 K (200°C). From these facts, plastic straining is affirmed to form vacancies. High cycle fatigue loading by rotating beam fatigue testing on martensitic steel is reported to create point defects, presumably vacancies, after substantial number of fatigue cycles. This was also concluded by hydrogen pick-up determined by TDA.

This knowledge assures that straining, monotonic or cyclic, definitely causes vacancy formation. More specifically, peak B in Figs. 3, 6 and 8 corresponds to vacancies. Severe plastic deformation like cold drawing is expected to cause vacancy cluster formation because of the accumulation of a high density of vacancies. Increased amount of vacancies has been revealed by positron life time measurement of specimens given straining after hydrogen charging. Increased amount of vacancies could lead to the vacancy cluster formation. Sub-peak C that is separated in Fig. 7(b) likely corresponds to vacancy clusters.

Binding energies of hydrogen with vacancy and vacancy cluster have been reported to be 46–51 kJ/mol and 68 kJ/mol respectively, suggesting that vacancy cluster is more stable, and its peak temperature locates at higher temperatures. Clustering of vacancies may proceed in different stages each having respective binding energies with hydrogen. It can explain the wider peak width for H100 in Table 4. These also assure that peak C corresponds to vacancy clusters.

As shown in Fig. 6(a), A1 peak height of #100 was almost same with that of #4% in Fig. 3(a), where the peak widths were same as 20 K. It means that the dislocation density is almost same between the two specimens. Nevertheless, the height of peak B (vacancy) in cyclically strained specimen #100 was much higher than that of monotonically strained #4%. A very high density of vacancies may be produced by cyclic straining. Essmann and Mughrabi observed dislocation substructure by transmission electron microscopy and rationalized the formation of high density vacancies in the case of fatigue loading, i.e. cyclic straining, through annihilation of dislocations with opposite signs.

When the specimen is hydrogen pre-charged before cyclic straining, the high density of vacancies can be stabilized by combination with hydrogen, just same as the case of H4%. Accordingly a great deal of vacancy cluster is encouraged to form. However, because of successive stages of clustering, the binding energy of hydrogen with vacancy clusters may scatter causing a large peak width (PW) of the peak C in Tables 4 and 5.

4. Discussion

4.1. New Hydrogen Traps Caused by Straining

A new kind of hydrogen traps is produced around of 385 K by slow tensile straining without hydrogen pre-charging. It is clearly shown as peak B in Fig. 3(a) or 4. Hydrogen charging before straining produces another peak C beyond 400 K. As those peak temperatures are higher than that of peak A1 or A2, they are suggestive of lattice defects whose binding energies with hydrogen are larger than those with dislocations or grain boundaries.

| Name | Item | A1 | A2 | B | C |
|------|------|----|----|---|---|
| BH100 | Height, ppm/min | 0.029 | 0.020 | 0.021 | 0.0045 |
|       | Temp., K | 333 | 368 | 393 | 418 |
|       | Width, K | 21 | 14 | 13 | 18 |
|       | Peak height ratio (A2/A1) | 0.67 |

Fig. 9. Comparison of peak height among #100, H100 and YH100. Not pre-charged but strained 100 cycles (#100), pre-charged with X3 condition and strained 100 cycles (H100) and pre-charged at Y10 condition. All specimens are post charged with X3 condition before TDA measurements.
strain amplitude produced new lattice defects that concern with vacancies and vacancy clusters. These lattice defects were built up by hydrogen pre-charging before straining. In TDA from specimens without hydrogen pre-charging, i.e. #4% in Fig. 4 and #100 in Fig. 7, the heights of peak A1 were clearly increased than that of #0, suggesting the increase in dislocation density by both tensile and cyclic straining; peak height of A2 grows in accordance with peak A1, i.e. the ratio of A2 to A1 is almost unaffected. When hydrogen was pre-charged, peak height of A2 of 0.04 tensile straining (H4% in Fig. 4) clearly increased in comparison with #4%. The hydrogen trapped by dislocations in H4% is calculated to be 0.296 mass ppm and 1.7 times larger than the amount 0.176 mass ppm for #4%. In the case of cyclic straining, as shown by #100 and H100 in Fig. 7, peak height of A2 was almost unaffected by hydrogen pre-charging. But it was clearly increased when hydrogen pre-charging was intensified as shown YH100 in Fig. 7. Straining under existence of hydrogen considerably increases the peak height of A2 that is the grain boundary traps, although the degree depends on the mode of straining. In the case of fracturing along grain boundaries, there is a discussion about hydrogen concentration along or in the vicinity of grain boundaries.17–19) The above-mentioned finding seems important.

5. Summary

Effects of cyclic straining on the development of lattice defects were studied in medium carbon steel containing globular cementite, using thermal desorption analysis (TDA) of post-charged hydrogen that acts as a tracer. Then the TDA curves were statistically analyzed using Gaussian function to separate the comprised sub-peaks. The influence of hydrogen pre-charging was further explored.

(1) TDA curves are deformed to have swelling at high temperature side by straining. It is emphasized by hydrogen pre-charging. These suggest the formation of strong hydrogen traps. They are more remarkable in the case of cyclic straining.

(2) TDA curves are decomposed to several desorption curves of hydrogen, assuming Gaussian function. The considered trap sites are dislocations, grain boundaries, vacancies and vacancy clusters. It enables quantitative comparisons of hydrogen traps.

(3) Tensile straining of 0.04 readily forms small amount of vacancies. One hundred cycles of 0.004 strain amplitude is more effective in vacancy formation, together with vacancy cluster formation.

(4) Hydrogen pre-charging before straining enhances the formation of those kinds of lattice defects. The maximum values of vacancies and vacancy clusters attained in the present study are 21.9% and 6.5% of the total desorption respectively.

(5) It is also revealed that hydrogen from dislocation traps increases by straining under hydrogen existence. Slow tensile straining is more notable than cyclic straining, while the latter is increased by intensified hydrogen pre-charging.

REFERENCES

1) K. Takai, G. Yamauchi, M. Nakamura and M. Nagumo: J. Jpn Inst. Met. Mater., 62 (1998), 267(in Japanese).
2) M. Nagumo, K. Ohta and H. Saitoh: Scr. Mater., 40 (1999), 313.
3) K. Takai, H. Shoda, H. Suzuki and M. Nagumo: Acta Mater., 56 (2008), 5158.
4) M. Nagumo, H. Shimura, T. Chaya, H. Hayashi and I. Ochiai: Mater. Sci. Eng. A, 348 (2003), 192.
5) I. Maroef, D. L. Olsen, M. Eberhart and G. R. Edwards: Int. Mater. Rev., 47 (2002), 191.
6) K. Takai: Trans. Jpn. Soc. Mech. Eng., A70 (2004), 9.
7) A. McNabb and P. K. Foster: Trans. AIME, 227 (1963), 618.
8) T. Yamaguchi and M. Nagumo: ISIJ Int., 43 (2003), 514.
9) K. Ebihara and H. Kaburaki: ISIJ Int., 52 (2012), 181.
10) T. Yokota and T. Shiraga: ISIJ Int., 43 (2003), 534.
11) T. Izumi and G. Itoh: Mater. Trans., 52 (2011), 130.
12) Y. Tsuichida: ISIJ Int., (in press).
13) K. Sakaki, T. Kawase, M. Hirato, M. Mizuno, H. Araki, Y. Shirai and M. Nagumo: Scr. Mater., 55 (2006), 1031.
14) M. Myers, S. T. Pieraux and R. E. Stoltz: J. Appl. Phys., 50 (1979), 5710.
15) M. Myers, M. M. Follstaedt, F. Besenbacher and J. Bottiger: J. Appl. Phys., 53 (1982), 8734.
16) U. Essmann and H. Huglebri: Philos. Mag. A, 40 (1979), 731.
17) K. Yoshino and C. J. McMahon, Jr.: Metall. Trans., 5 (1974), 363.
18) M. Yamaguchi, K. Ebihara, M. Itakura, T. Kadoyoshi, T. Suzudo and H. Kaburaki: Metall. Mater. Trans., 42A (2011), 330.
19) M. Nagumo and H. Matsuda: Philos. Mag. A, 82 (2002), 3415.