Prior Austenite Grain Boundary Detection in an Ultra-Cleaned Cr Bearing Microalloyed Steel

Seyed Sadegh GHASEMI BANADKOUKI* and Misagh GHADIRI ANARI
Department of Mining and Metallurgical Engineering, Yazd University, Safayieh, Daneshgah Blvd, University Main Campus, P. O. Box: 89195-741 Yazd, Iran.

(Received on August 16, 2017; accepted on June 14, 2018)

In this study, several single chemical and double thermal-chemical etching techniques have been used in order to identify in details the prior austenite grain boundaries (PAGBs) with a good contrasting resolution in an ultra cleaned high strength low alloy Cr bearing microalloyed steel. The general chemical etching techniques are based on the various conventional metallographic reagents involving: Picral; 60°C hot Picral; and Vilella solutions; while the double stage of thermal-chemical procedures have been classified into 400°C low and 650°C high temperature thermally treated for 3 hours followed with general chemical etching. Light and scanning electron microscopic observations have been supplemented with energy dispersive X-ray spectroscopy to follow the PAGBs detection in conjunction with that of carbide precipitation. Evidences are presented which indicate that only a tracing detection of PAGBs can be identified with general single metallography using conventional chemical etching reagents, while the double stage of thermal-chemical etching treated samples are associated with sharp contrasting resolution of PAGB areas. These observations are rationalized in terms of higher stimulation of chromium rich carbide formation and consequently the higher dilution of solute chromium at grain boundary areas of thermal-chemical treated samples. The PAGBs are considerably enriched from solute chromium in the direct water quenched fresh martensitic samples, while the thermal-chemical treated samples have been associated with chromium rich carbide precipitation at grain boundaries causing a higher sensitivity of grain boundaries to general chemical etching solutions, resulting the higher contrasting resolution of grain boundary areas.

KEY WORDS: ultra-cleaned microalloyed steel; prior austenite grain boundaries; general chemical metallography; double thermal-chemical metallography.

1. Introduction

In a low alloy heat treatable steel, it is obvious that the characteristics of phase transformations and microstructures are significantly affected by the prior austenite grain size developed before cooling, and consequently the mechanical properties are quite variable depending on the steel chemical composition and heat treatment schedule.1–8) To investigate the separate effect of prior austenite grain size on the microstructures and mechanical properties of low alloy heat treatable steels, detection of prior austenite grain boundaries (PAGBs) with sufficient contrasting resolution is one of the most important research areas in physical metallurgy and focused by numerous investigators.9–18) In this regard, several metallographic techniques have been developed in order to illustrate PAGBs in low alloy heat treatable steels and the most common metallographic techniques can be classified as either general single stage of chemical metallography or high temperature thermally preparation procedures.3–7,17,18,27,28) The general chemical etching has been traditionally used to detect the PAGBs because the associated experimental method is much simpler rather than that of other procedures.7–9,11) The earliest chemical etching is based on the Vilella chemical reagent which has been developed in 1938 and used with some success for revealing PAGBs of conventional medium to high carbon martensitic steels.18) Some years later, the first reasonably successful to chemical etchant for revealing austenite grain boundaries has been occurred by Bechet and Beaujard, who have used a saturated aqueous picric acid solution containing 0.5% of a wetting agent such as sodium alkylsulfonate.21) This etchant has been widely used to illustrate PAGBs of medium to high carbon martensitic steels when those steels have been tempered below that of 565°C. Several investigators have also reported that the PAGBs can be nicely revealed in low alloy heat treatable steels containing some impurity elements particularly if the martensitic specimens have been heated at a temperature in the tempering embrittlement range.22–27) Pre-ece and Cartooneshow have also shown that there is a clear appearance in PAGBs when the phosphorous containing low alloy martensitic steels have been tempered because of higher localized phosphorous segregation at grain boundary areas.23) However, the segregation of Sn or Sb to PAGBs, which have been associated with tempering embrittlement, does not help to reveal the PAGBs using Picral reagent in
low phosphorus low alloy steels. Vander Voort has developed the most successful Picral chemical solution containing a wetting agent, usually sodium dodecylbenzene sulfonate, which can be used at room temperature for a period of 4–20 minutes etching time. Meanwhile, this etchant has not been suitable to reveal the PAGBs in martensitic low alloy steels with carbon concentration below that of about 0.3%. These arguments are still underway and there is not a general single chemical etching to detect PAGBs of ultra-cleaned high strength low alloy steels. Furthermore, it has been also reported that the PAGBs can be visualised by various high temperature thermal preparation procedures. The thermally treated techniques are in association with revealing directly PAGBs in a finely prepolished specimen surface by formation of grooves at austenite grain boundaries by surface tension effects and matter transport mechanisms when the specimens have been exposed to a high temperature thermally treated condition in an inert atmosphere. These grooves make the austenite grain boundaries visible in light microscopy. However, at a high temperature of austenitizing heat treatment, the grooves that reveal the austenite grain boundaries can be interfered with the advance of austenite grain boundaries, producing some spasmodic migration of grain boundaries on the prepolished specimen surface and also the presence of some ghost traces at grain boundary areas. Therefore, the procedure for detecting PAGBs is quite variable and should be investigated in case to case study depending on the steel chemical composition, the level of impurity elements, the heat treatment cycles and also some of the other unidentified factors. This process has been even a more difficult task, especially in the ultra cleaned high strength low alloy steels in which the associated PAGBs are low sensitive to chemical etching reagents. A considerable effort should be extended to develop the best metallographic procedure for detecting of PAGBs in a certain steel chemical composition. In this study, several single chemical etching and double stage thermal-chemical metallographic techniques have been used to detect the PAGBs in an ultra cleaned high strength low alloy Cr bearing microalloyed steel.

2. Materials and Experimental Procedures

The chemical composition of investigated ultra cleaned Cr bearing high strength low alloy steel was presented in Table 1. All of the proposed heat treated samples were cut from the as-received 20 mm diameter steel rod and were first normalized after being heated at 900°C for 60 minutes to achieve a more homogenized starting microstructure in the samples. Then, the samples were reaustenitized at 900°C for 60 minutes followed with water quenching to keep the PAGBs at room temperature. The mechanical grinding and polishing procedures were carried out according to ASTM E3 standard. In order to reveal the PAGBs with good contrasting resolution, three sets of pretreated samples were investigated according to the following single chemical and double thermal-chemical metallographic techniques.

2.1. General Chemical Metallography

In this condition, a set of direct water quenched fresh martensitic samples was finely polished and etched using various general chemical reagents as listed in Table 2.

2.2. Double Thermal-Chemical Metallography

2.2.1. Low Temperature Thermal-Chemical Metallography

In this method, the selected fresh martensitic samples were reheated at 400°C for 3 hours to sensitize PAGBs to impurity segregation, followed by air cooling to room temperature. The polishing and etching procedures were carried out according to ASTM E3 standard and general chemical reagents listed in Table 2, respectively.

2.2.2. High Temperature Thermal-Chemical Metallography

In this route, the proposed fresh martensitic samples were reheated at 650°C for 3 hours to achieve chromium rich carbide precipitation at PAGB areas, air cooled to room temperature, and then followed with polishing and etching procedures in the same fashion as to the low temperature of 400°C thermal-chemical treated samples.

The observations of PAGBs were carried out under both of light and electron microscopes. For comparing the level of impurity concentration at PAGBs, the analysis of chemical composition was carried out at various locations of PAGB areas with energy dispersive X-ray spectroscopy (EDX) technique. The EDX analysis was widely applied at various locations of PAGBs to identify the impurity concentration in conjunction with that of carbide precipitation using a scanning electron microscopy (SEM) model TESCAN-VAGA-II operated at an accelerating voltage of 15 kV.

3. Results and Discussion

3.1. General Chemical Metallography

Figure 1 shows typical light micrographs of best quality possible for PAGBs detection using general chemical etching solutions listed in Table 2. For Picral reagent, the martensitic microstructure has been initially started to appear after about 10 minutes, while the PAGBs are not sensitive to the chemical reagent. For keeping a longer period of etching

Table 1. Chemical composition of the investigated Cr bearing microalloyed steel (in weight percent).

| C   | Si | Mn | P  | Cr | Ni | Cu | N  | Nb | Ti | Fe |
|-----|----|----|----|----|----|----|----|----|----|----|
| 0.2 | 0.1| 1.5| 0.003 | 0.013 | 1.98 | 0.25 | 0.34 | 0.0091 | 0.017 | 0.013 | Balance |

Table 2. General chemical etching reagents employed in this experimental work.

| Etching reagent | Chemical composition                                      |
|-----------------|-----------------------------------------------------------|
| Picral          | 5 gr picric acid + 100 ml ethanol alcohol                  |
| 60°C hot Picral | 5 gr picric acid + 100 ml ethanol alcohol at 60°C          |
| Vilella         | 1 gr picric acid + 5 ml HCl + 95 ml ethanol alcohol        |

© 2018 ISIJ
time up to 15 minutes, the metallographic specimen surfaces have been darkened with a slight detection of austenite grain boundary as indicated by arrowed in the micrograph (Fig. 1(a)). It is interesting to emphasize that the use of Picral etchant solution has been related with a severe darkening of martensitic microstructure in just a few minutes, and further holding time has been not resulted to the higher contrasting resolution of PAGBs in the micrographs. For this reason, it has been tried out by using hot Picral solution at 60°C to enhance the corrosive behavior of etching reagent, and consequently increasing the higher sensitivity of PAGBs to the chemical reagent. Figures 1(b) and 1(c) are the associated light micrographs for the best quality evidences of such successes for PAGBs, indicating that the higher pitting and darkening of metallographic specimen surfaces have been developed after using 60°C hot Picral reagent for shorter etching time of 9 minutes in comparison to that of 15 minutes etched samples in room temperature general Picral solution. On the other hand, when the samples have been etched in 60°C hot Picral solution, very small bubbles have been formed and consequently cracked down at just near the metallographic specimen surfaces, resulted a lower contrasting resolution of PAGBs in the microstructures. Moreover, the use of hot Picral solution has been occurred with some more toxic vapors which can be undesirable for health as well.

The first positive result of PAGBs detection has been obtained after using general chemical etching technique based on the Vilella reagent as shown in Fig. 1(d). In this condition, a sharp contrasting resolution of martensitic matrix phase in conjunction with a tracing location of PAGBs have been revealed after a few second of etching times, as indicated by arrows in the micrograph.

From the light micrographs presented in Fig. 1, it is evident that only a tracing detection of PAGBs can be obtained after using Vilella chemical solution in comparison to that of either room temperature Picral and/or 60°C hot Picral reagents. These results indicate that the use of various single metallographic techniques based on the conventional chemical reagents are not suitable for revealing the PAGBs in this ultra cleaned Cr bearing microalloyed steel. This claim can be fully supported according to the relatively high level of 1.98% solute chromium in conjunction with that of low concentration of impurity elements such as sulfur (S) and phosphorous (P) in the direct water quenched fresh martensitic samples. On the other hand, according to the quantometry analysis of this microalloyed steel presented in Table 1, it is obvious that the amounts of S and P are very low in the chemical composition, and consequently it is reasonable to conclude that the accumulation of S and P rich nonmetallic compounds are almost negligible at PAGB areas. Furthermore, the relatively high level of 1.98%Cr has been totally soluble in the fresh direct water quenched martensitic microstructures, resulted in a lower sensitivity of austenite grain boundary to general chemical etching reagents. Consistent with these claims, electron microscopic investigation has been also illustrated that the impurity free austenite grain boundary formation has been developed in water quenched fresh martensitic samples. For this reason, Fig. 2 shows a scanning electron microscopic micrograph of prior austenite grains in conjunction with a typical EDX analysis taken from a deeply etched fresh martensitic
sample. The location of EDX analysis was the indicated triple point of PAGB area shown by an open circle marked with A symbol, illustrating that there are not S and P in the chemical composition of grain boundary areas. This might be a reason why the general chemical etching reagents listed in Table 2, which are sharply attacked to PAGBs of traditionally developed medium carbon heat treatable martensitic steels, are not sensitive to this ultra cleaned microalloyed steel. On the other hand, it is obvious that the martensitic phase transformation has been occurred with burst nucleation and growth of martensitic crystal within a particular prior austenite grain and that the martensitic crystal growth has been stopped at the prior austenite grain boundary, causing higher energy regions at area close to the prior austenite grain boundaries in comparison to that of low angle martensitic crystals formed within the austenite grains. Therefore, the PAGBs can be revealed with good contrasting resolution in the secondary electron micrographs taken after highly deep etching of the water quenched fresh martensitic samples, while the light optical micrographs are completely darkened in this case. These results indicate that the detection of PAGBs has been not developed with general chemical metallography under light microscopic observation of investigated microalloyed steel, and so several thermally treatments should be employed in order to increase the sensitivity of PAGBs to chemical reagents.

3.2. Double Thermal-Chemical Metallography

Figure 3 shows typical light micrographs of prior austenite grains taken from the samples treated with double stage of metallography based on the thermally treatment followed with general chemical etching. Figure 3(a) illustrates PAGBs in the low temperature of 400°C thermally - treated samples for 3 hours followed with 6 seconds Villela chemical etching, while Fig. 3(b) is in association with that of higher sensitized temperature of 650°C in the same thermal and chemical condition. These observations indicate that the progress of PAGBs detection is quite variable depending on the temperature of thermally activated condition. The PAGBs are sharply contrasted in the light micrographs taken from the samples obtained at higher temperature of 650°C thermal-chemical treatment in comparison to that of lower temperature of 400°C thermal-chemical preparation ones.

On the other hand, the micrograph of prior austenite grains taken from the lower thermally sensitized samples followed by chemical etching is not clear in some areas of microstructures, while in the higher temperature thermal-chemical treated samples, the PAGBs are sharply contrasted in the
microstructures. These results can be also rationalized with low concentration of solute chromium at PAGB areas which can be occurred as a consequence of chromium rich carbide precipitation during thermally activated treatment. In this regard, Fig. 4 illustrates a typical SEM micrograph of 650°C thermal-chemical treated samples in conjunction with two locations marked as A (grain boundary carbide particle) and B (PAGB area) arrows at which spot EDX analyses have been carried out, and the relevant results are indicated in Figs. 4(b) and 4(c), respectively. For more comparison, Table 3 also repeats the EDX concentration results of carbon and other alloying elements within the carbide particle and adjacent PAGB area, according to the locations marked with A and B arrows in the electron micrograph presented in Fig. 4(a). It is interesting to emphasize that although the measurement of carbon concentration within chromium rich carbide precipitate and adjacent PAGB area by EDX analysis technique has been accompanied with some more overestimation, this technique can be used as a comparative study in order to identify the variation of carbon concentration within various microphases developed in low alloy steels.31,32) Comparison of the above EDX results indicates that the chromium rich carbide formation has been occurred with more chromium and also higher carbon partitioning in comparison with that of lower concentrated PAGB areas during soaking treatment at 650°C for 3 hours in the thermal-chemical treated samples.

A possible explanation for higher sensitivity of austenite grain boundary to general chemical etching can be made according to the higher carbide precipitation theory at grain boundary areas of higher temperature thermal-chemical treated samples (Figs. 3, 4). It is a well known fact that the diffusion of substitutional alloying elements such as iron and chromium can be accelerated in the higher temperature of 650°C thermally treated samples than that of lower temperature of 400°C ones. The carbide precipitation is much more easy to be formed at a higher temperature of thermally treated in the higher energy grain boundary locations. Thus, a considerable higher level of chromium rich carbide precipitation can be developed at grain boundaries of higher temperature thermal-chemical treated samples, causing a higher dilution of solute chromium of grain boundary areas. Furthermore, a more chromium rich carbide precipitation can be associated with a higher energy of carbide/martensite interfaces, resulted in a higher sensitivity of grain boundary areas to general chemical etching as a consequence of more corrosion of grain boundary areas which can be developed in the same fashion as to the general intergranular corrosion phenomenon observed in precipitation hardened metallic materials. This can be a reasonable explanation why the sensitivity of PAGBs to general chemical etching has been significantly increased in the higher temperature of 650°C thermally treated samples, and so the higher contrasting resolution of grain boundary areas can be obtained by chromium rich carbide precipitation.

It has been reported by several investigators19,20) that some of the conventional chemical reagents are more sen-

| Location | Fe  | C   | Cr  | Mn  | Si  | Nb  | Ti  | Total |
|----------|-----|-----|-----|-----|-----|-----|-----|-------|
| A        | 54.51 | 16.37 | 29.12 | –   | –   | –   | –   | 100   |
| B        | 91.43 | 4.66  | 1.30  | 1.47 | 0.09 | 0.02 | 0.03 | 100   |


Fig. 4. SEM micrograph with the associated EDX analyses of carbide precipitate (A) and the adjacent PAGB area (B) taken from the high temperature of 650°C thermal-chemical treated samples followed by 57 seconds etching in Vilella reagent showing: (a) the general feature of carbide formation at grain boundary areas with the locations of EDX; (b) and (c) the EDX analyses of chromium rich carbide particle and adjacent PAGB location marked with A and B arrows in (a), respectively.
sitive to PAGBs of medium carbon low alloy tempered martensitic steels, because the tempering heat treatment has been associated to segregation of some impurities at grain boundaries. This phenomenon is consistent with our experimental work regarding that reheating of fresh martensitic samples in tempering temperature range has been associated with chromium rich carbide precipitation at grain boundary areas. Electron microscopic investigation indicates that the thermal-chemical treated samples have been in association with chromium rich carbide precipitation, and so the austenite grain boundaries are significantly diluted from solute chromium, causing a higher sensitivity of PAGBs to conventional chemical etching solutions as a consequence of lower corrosion resistance of grain boundary areas. Inversely, in the direct water quenched fresh martensitic samples, the progress of grain boundary carbide precipitation is almost negligible, and so when the samples are etched the carbide free PAGB areas are not sensitive to general chemical reagents, causing much lower contrasting resolution of grain boundaries in the light micrographs (Figs. 1, 3).

It is interesting to point out that in the double stage of metallography based on the thermally treatment followed with conventional chemical etching used in the present investigation, the higher chromium rich carbide accumulation and consequently the higher dilution of solute chromium at grain boundaries have been associated to higher contrasting resolution of PAGBs in the microstructures (Fig. 4). The higher contrasting of PAGB has been developed for higher temperature of 650°C thermal-chemical treated samples in comparison to that of either lower temperature of 400°C thermal-chemical treatment and/or the fresh martensitic samples (Figs. 1, 3). These results are inconsistent with some of the previous investigator studies who have demonstrated that double metallographic techniques based on the combination of thermal and chemical etching are not successful to illustrate PAGBs of medium carbon microalloyed steels and that the PAGBs of ultra cleaned high strength low alloy steels can be only detected by reaustenitizing a fine prepolished sample in an inert atmosphere by formation of grooves at grain boundary areas. Therefore, it is reasonable to conclude that the chromium enriched M₃C type carbide precipitation has been formed in the 650°C thermal-chemical treated samples.

from 720°C to ambient temperature in microalloyed steel containing lower than 2%Cr in conjunction with 0.2%C. This means that the formation of chromium enriched M₃C type carbide precipitation can be accelerated with higher diffusion of heavy substitutional Cr atoms from martensitic matrix into the PAGBs because of higher thermally activated Cr atoms diffusion at higher temperature of 650°C. Therefore, it is reasonable to conclude that the chromium enriched M₃C type carbide precipitation has been formed in the 650°C thermal-chemical treated samples.

4. Conclusions

Detection of PAGBs in an ultra cleaned Cr bearing microalloyed steel has been widely investigated using several single chemical and double thermal-chemical etching techniques. The main conclusions are presented as follows:

(1) The general single metallographic techniques based on the conventional chemical solutions involving: Picral; 60°C hot Picral; and Vilella reagents are unsuccessful to reveal the PAGBs of ultra cleaned Cr bearing microalloyed steel. Only, a tracing detection of PAGBs has been developed after using Vilella chemical etching solution.

(2) Detection of PAGBs is sensitive to either impurity segregation and/or carbide precipitation at grain boundary areas. A thermally sensitive heat treatment involving reheating of fresh martensitic samples in the tempering temperature range followed with general chemical etching can be used to reveal the PAGBs of ultra cleaned Cr bearing microalloyed steel.

(3) The high temperature of 650°C thermal-chemical etching samples have been associated with chromium rich carbide precipitation at grain boundary areas. The higher energy of carbide/martensite interfaces in conjunction with that of higher dilution of solute chromium developed as a
consequence of carbide formation are related to the higher sensitivity of grain boundaries to general chemical etching reagents, resulting much higher contrasting resolution of PAGBs in the light micrographs.

(4) The combination of thermally treatment followed by chemical etching is an effective method for revealing the PAGBs in the ultra cleaned Cr containing microalloyed steel. The higher temperature of 650°C thermally treated followed with Vilella chemical etching has been associated to higher chromium rich carbide precipitation at grain boundaries, causing a higher contrasting resolution of PAGB areas.

(5) The higher contrasting resolution for simultaneous development of PAGBs in the investigated ultra cleaned microalloyed steel has been increased in the manner of: Picral; 60°C hot Picral; Vilella; low temperature of 400°C thermal-chemical etching; and high temperature of 650°C thermally treated followed with subsequent using of Vilella chemical etching.

REFERENCES
1) G. Krauss: Steels Heat Treatment and Processing Principles, ASM International, Materials Park, OH, (2005), 179.
2) J. Seok and K. Young: Mater. Des., 29 (2008), 1840.
3) A. K. Giumelli, M. Militzer and E. B. Hawbolt: ISIJ Int., 39 (1999), 80.
4) S. Jiao, J. Penning, F. Leysen, Y. Houbaert and E. Aernoudt: ISIJ Int., 40 (2000), 1035.
5) T. Reti, Z. Fried and I. Felde: Comput. Mater. Sci., 22 (2001), 261.
6) S. C. Hong, S. H. Lim, H. S. Hong, K. J. Lee, D. H. Shin and K. S. Lee: Mater. Sci. Eng. A, 355 (2003), 241.
7) A. K. Alogab, D. K. Matlock, J. G. Speer and H. J. Kleebe: ISIJ Int., 47 (2007), 307.
8) A. K. Alogab, D. K. Matlock, J. G. Speer and H. J. Kleebe: ISIJ Int., 47 (2007), 1034.
9) K. S. Cho, H. S. Sim, J. H. Choi, K. B. Lee, H. R. Yang and H. Kwon: Mater. Charact., 59 (2008), 786.
10) S. Markopoulos, S. Karagiannis and N. Ridley: Mater. Sci. Eng. A, 483 (2008), 735.
11) D. San Martin, P. E. J. Rivera Diaz Del Castillo, E. Peekstok and S. Vander Zwaag: Mater. Charact., 58 (2007), 455.
12) H. Seok Yang and H. K. D. H. Bahadeshia: Scr. Mater., 60 (2009), 493.
13) J. Seok and K. Young: MSc. Thesis, Yenisei University, (2005), 120.
14) A. H. Meysami, R. Ghasemzadeh, S. H. Seyedian and M. R. Aboutalebi: Mater. Des., 31 (2009), 1570.
15) K. O. Lee, S. K. Hong, Y. K. Kang, H. J. Yoon and S. S. Kang: Int. J. Auto. Tech., 10 (2009), 697.
16) H. Kwon, J. C. Cha and C. H. Kim: Mater. Sci. Eng., 10 (1988), 121.
17) J. Hornikova, P. Sandera and J. Pokułda: Journal of ASTM International, 2, (2005), 53.
18) S. Kim, S. Lee and B. S. Lee: Mater. Sci. Eng. A, 359 (2003), 198.
19) C. Garcia de Andres, M. J. Bartolome, C. Capdevila, D. S. Martin, F. G. Caballero and V. Lopez: Mater. Charact., 46 (2001), 389.
20) J. R. Vilella: Metallographic Technique for Steel, American Society for Metals, Cleveland, OH, (1938), 84.
21) S. Bechet and L. Beaujard: Rev. Met., 52 (1955), 830.
22) A. H. Ucisk, H. C. Feng and C. J. Memahon: Metall. Trans., 9 (1978), 321.
23) A. K. Cianelli, H. C. Feng, A. H. Ucisik and C. J. McMahon: Metall. Trans. A, 8 (1977), 1059.
24) A. H. Ucisk, C. J. Memahon and H. C. Feng: Metall. Trans., 9 (1978), 604.
25) A. Preece and R. D. Carter: J. Iron Steel Inst., 173 (1953), 387.
26) J. A. Nelson: Prakt. Metallogr., 4 (1967), 192.
27) G. F. Vander Voort: Mater. Charact., 35 (1995), 135.
28) G. F. Vander Voort: Metallography: Principles and Practice, ASM International, Metals Park, OH, (1999), 222.
29) C. Garcia de Andres, F. G. Caballero, C. Capdevila and D. San Martin: Mater. Charact., 49 (2003), 121.
30) E. Rabkin and L. Klinger: Mater. Sci. Technol., 17 (2001), 772.
31) S. S. Ghasemi Banadkouki and E. Fereiduni: Mater. Sci. Eng. A, 619 (2014), 129.
32) C. Y. Wang, J. Shi, W. Q. Cao and H. Dong: Mater. Sci. Eng. A, 527 (2010), 3442.
33) W. F. Smith: Structure and Properties of Engineering Alloys, McGraw-Hill, New York, (1994), 290.