Effect of Scandium on Homogenization Treatment and Friction Stir Processing on Mechanical Properties of 7075 Aluminium Alloy Prepared by Foundry Route

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Abstract

The aluminium alloy (7075 series) with minor scandium addition prepared by foundry route, was subjected to homogenization treatment at different temperature from 300 to 500°C for 8h and also multi-pass friction stir processing (MP-FSP) was performed to improve surface properties of cast aluminium alloy. The effect of minor scandium (0.33wt.% ) on the microstructure and mechanical properties of prepared alloy was investigated using optical microscopy, SEM, FESEM, XRD, TEM, Vicker’s hardness and tensile test. The homogenized microstructure was obtained showing fine grains because homogenization of cast structure and dispersion of second-phase particles also occur. The Al-Zn-Mg alloy with high strength, good corrosion resistance and good welding property is used widely in various industrial applications. In addition of Sc to increase the strength mainly comes from fine grain strengthening, substructure strengthening and precipitation strengthening caused by Al3(Sc) and MgZn2 particles. These particles are formed during homogenization treatment of cast aluminium alloy and it has great influence on precipitation behaviour of Al3(Sc) particles.

Key words: 7075 aluminium alloy; homogenization treatment; grain refining strengthening; MP-FSP.

1. Introduction

The aluminium alloys (7075 series) have greatest age-hardening response due to their excellent combination of low density and high strength make them very attractive materials in aerospace, automotive, and marine industries [1-3]. The microstructures and properties of aluminium alloys are strongly affected by adding small quantities of scandium (Sc). Significantly grain refinement in castings are obtained by promoting heterogeneous nucleation sites in the melt, which may constitutes the basis for grain refinement [4-6]. Sc forms a high melting eutectic phase with aluminium at 655°C of 0.55wt.%Sc, Al3Sc(L12-type) phase and lattice parameter close to that of aluminium [7-10]. Fine-grained of 7075 Al alloy (0.33% Sc) has an average grain size of 23.0±2.15 µm, when lattice constant of Al3Sc particle (FCC) is similar to α(Al), and disregistry about 1.34% at ambient temperature, assuming no residual stresses and taking lattice parameter values of 0.4049 nm and 0.4103 nm for Al and Al3Sc, respectively [11-15]. These inoculated particles have high melting point like TiB2, TiC, AlB2 etc. Especially the anti-recrystallizing effect is the most pronounced among all the alloying elements in aluminium due to the Al3Sc (L12-type) dispersoids which are thermally stable and remain insoluble at high temperatures [16, 17]. The alloy is fully homogenized when the interior volume of second phases (η, β, T etc.) are minimized and numerous fine Al3Sc dispersoids (1-50 nm size) across the grain. These second phases are undesirable because they diminish the mechanical properties for nucleation sites of recrystallization. While the dispersoids are desired to pin the grain boundaries, inhibiting recrystallization and refine the grains at 300-500°C [18-20]. Fe and Si are the most common impurities also negatively affect the hot workability which should be spheroidized by homogenization treatment activated through atomic diffusion phenomena inside the grains. The solute atoms diffuse from higher concentration to lower concentration at the grain boundary to the grain till composition become uniform. It can be expressed by the dynamic equation of homogenization as follows: \( \frac{1}{T} = \frac{P}{R} \ln \left( \frac{t}{Q} \right) \), where T for homogenization temperature (K), t for homogenization time (h), L for dendrite arm space (µm), P = R/Q, R for gas constant (J/mol.K), and Q = 4.6/4π2Do2, Do for diffusion coefficient (cm²/S) and not related to the temperature. It can be concluded that when the homogenization temperature is constant, the time is shortened as the dendrite
space reduces, meaning the refinement of cast structure can improve the diffusion speed of solute atoms during homogenization treatment. In addition, there is a good correlation between boundaries and dispersoids and it can express by Zener drag effect: 

\[ P_z = \frac{3\gamma r}{V_f} \]  

where \( V_f \) for volume fraction of the dispersoids, \( r \)-particles radius, and \( \gamma \)-energy for the boundary layer. At a critical ratio \( \frac{V_f}{r} \), when the Zener drag important to overcome the driving force for boundary migration that delays the recrystallization process [21, 22]. Thus, the smallest grain size (13.79±2.89 µm) can be achieved through homogenization treatment at 450°C for 6h due to the strongest recrystallization inhibition of the presence of numerous dispersoids. In addition, the MP-FSP is a surface modification technique for cast aluminium alloy to refine coarse particles, heal the casting porosity, and consequently increase strength. During MP-FSP, a specially designed cylindrical tool is plunged into the plate causing intense plastic deformation through stirring action, yielding a defect free, and dynamically recrystallized, fine grained microstructure. The resulting grain size varies from microns to few nanosized after MS-FSP [23-25]. The cast plate size of 150x90x8 mm³ was selected for this investigation. The MP-FSP zone 150 mm long was produced at a tool travel speed (70 mm/ min) and a rotational speed (1000 rpm) and axial force (15kN). It has been reported that the chemical composition, production technology and heat treatment, all affect the precipitation processes as well as amount of strengthening [26-28]. In this paper, the relationship between the mechanical behaviour and microstructure characteristics of 7075 aluminium alloy with minor Sc has investigated to understand the mechanism of grain refinement and dispersive precipitation after homogenization treatments and as well as MP-FSP.

2. Materials and Experimental Procedure

The alloy was prepared by foundry route using high purity Al, Zn, Mg and Al-2wt.%Sc master alloy. Al is a light weight (\( \rho \), 2.72 gm/cc) metal and high thermal conductivity of 167 W/mm-K. Sc is a costlier element and high melting point of 1539°C and 3d transition metal. A muffle furnace was used to melt the aluminium alloy through foundry route. A mild steel mould was used a rectangular size (200×90×24 mm³). The charge consists of small Al pieces which kept into a graphite crucible. Sc was added in the melt in-form of Al-2wt.% Sc master alloy. The melting procedure was done as the following steps such as aluminium pieces (3.0 kgs) heated up to the 780°C for 2.5h in graphite crucible which placed in a muffle furnace, then master alloy was added in the proportion (generally 50% recovery) to melt just half an hour before pouring into the mild steel mould. Then, the hot graphite crucible was taken outside from the muffle furnace and gradually added Mg and Zn into the melt with carefully control fading effect. The slag was removed from the top of the melt and its poured into mould quickly and dipped into water for faster cooling. In foundry practice aluminium alloys are grain refined in the liquid state before solidification of casting.

The wet analyses (ICP-MS and AAS methods) of as-cast alloy gave the following average composition (wt.%): Al-6.47%Zn-1.54%Mg-0.33%Sc-0.19%Si-0.21%Fe. The metallographic samples were polished by mechanical polisher to obtain a mirror finish images of Figure 2(b), Figure 4, and Figure 10, respectively. The etchant used was a Keller’s reagent (1ml HF+1.5ml HCl+2.5ml HNO₃+95ml H₂O) for this alloy. All optical images were taken by LEICA DMI 5000M microscope, and grain sizes were measured by Image J software. The sample (10×10×10 mm³) for homogenization treatment of aluminium alloy as selected temperatures are likely to 300°C for 8h, 350°C for 8h, 400°C for 8h, 450°C for 8h, and 500°C for 8h, respectively. The optical microstructures of homogenized samples are shown in Figure 4. The Vicker’s hardness (15kg. load) (FIE VM50 PC) values were taken randomly on the homogenization samples at the series of temperatures of 300, 350, 400, 450, and 500°C, respectively. At same time the sequences of 2, 4, 6, and 8h intervals thoroughly maintained for average of six indentations of hardness measurement and curves are drawn (Figure 5). The bar diagrams displayed peak hardness values at different homogenization temperatures from 300-500°C at 6h time (Figure 6). The FESEM microstructure with EDAX analysis was examined by light microscopy (QUANTA 200F, FEG LaB₆, 30kV) as shown in Figure 7. The XRD analysis was carried out for as-cast Al alloy (Bruker AXS diffractometer D8 Germany equipped with Cu Ka
radiation) as shown in Figure 8. The TEM samples were prepared through gently mechanically polishing then preferred for electrolytically etching (solution of 75% CH3OH + 25%HNO3, 12V, and -35°C) to make them thinner for final TEM and EDS (Techai G² 20S-TWIN at 200 kV) observations as shown in Figure 9. The SEM fractographic analysis (LEO 435 VP) was carried out for MP-FSP samples after tensile testing as shown in Figure 11.

Figure 3(a-c) shows a schematic representation of FSP in the present experimental work. This work examine the effect of MP-FSP on aluminium plate (150×90×8 mm³) by adding one pass at a time to the total of 15 passes as maintained in the following parameters (Table 1). The important parameters for repetitive MS-FSP tool rotational speed and transverse speed, which contributes to the heat input to refine microstructures of each successive passes at the same direction on the workpiece. A specified tool was rotated clockwise at the speed of 1000 rpm with the rotating pin inserted into the workpiece. In addition, there are three forces acting during MP-FSP likely to the axial force (Fz-direction) was held perpendicular to the workpiece. This force affect the temperature and hydrostatic pressure which are responsible for producing defects free processed zone. The direction of plate travel to the longitudinally (Fx-direction) acting force is called translational force on the plate. Another force is represented to the transversely (Fy-direction) to the workpiece. The complete flow chart for aluminium alloy preparation and MP-FSP conducted as per predetermined flow chart as shown in Figure 1. The tensile tests were carried out by using an Instron testing machine (25 kN, H25 K-S, UK) at a cross head speed of 1 mm/min at room temperature. Tensile samples were machined after collection from the MP-FSP zone. The measurements of tensile specimen is shown in Figure 3(c) and results are tabulated in Table 2.

Figure 1: Complete flow chart for aluminium alloy preparation and MP-FSP.

Figure 2: (a) The Al-Sc phase diagram, (b) The optical microstructure of as-cast 7075 Al alloy.
Figure 3: (a) Illustration of different forces acting during processing and FSP set-up, (b) Tool design, (c) Tensile test sample (ASTM: E8/E8M-11).

Table 1: Processing conditions of MP-FSP.

| Items                        | MS-FSP parameters                                                                 |
|------------------------------|-----------------------------------------------------------------------------------|
| Tool configuration           | shoulder diameter 25 mm, pin diameter 5 mm, pin height 3.5 mm, and made of harden martensitic stainless steel |
| Processing parameters        | tool rotation speed 1000 rpm, plate travel speed 70 mm/min, downward axial force 15 kN, 2° tilt angle |
| Processing direction         | clockwise and unidirectional, size of plate 150×90×8 mm³                           |
| Processing conditions        | 35% overlap from the first pass to second successive passes, 5 min rest in between two successive passes, and up to 15 passes. |

3. Results and Discussion

The effect of minor Sc addition will of course depend on the composition of the alloy. The additions of Sc in aluminium alloy appear to reduce hot tearing, shrinkage and porosities. As the Sc content increases within the specified solubility in the solid solution, the grains virtually do not disintegrate [29-31]. The maximum solubility of Sc exhibits 0.35 wt.% in Al-Sc binary phase diagram. When the amount of Sc exceeds the eutectic content and, consequently, there appear primary particle of the Al₃Sc phase, the grains disintegrate to the finest possible size. In addition, fine grain promotes the flow of molten metal to feed shrinkage during solidification, resulting in smaller and more uniform dispersed shrinkage or gas porosities. Fine grains also provide a complex network of grain boundaries, reducing tendency for hot crack initiation and propagation. There are three main reasons for adding Sc to aluminium alloys. All of these effects are related to the formation of particles of Al₃Sc, the phase that is in equilibrium with Al, precipitation of Al₃Sc may also take place at the temperatures used for homogenization of common heat treatable aluminium alloy. The Al₃Sc particles formed under these conditions, normally termed dispersoids, will usually be low density (>0.25 wt.%) for giving a strong contribution to the alloy strength [32, 33]. Two significant metallurgical principles are important such as Zn/Mg>2, and an overall reduction in saturation of the composition with respect to the theoretical maximum solubility [34]. In addition, MP-FSP is a novel solid state technique which provides microstructural refinement, and better mechanical properties of the processed zone. It can be precisely controlled by process parameters and specified tool design, and materials [35, 36]. The basic concept of MP-FSP has to be discussed for this experimental works. The MP-FSP was processed at a time interval of 5 minutes after each pass. This process was performed using 1000 rpm and 70 mm/min traverse speed for 15 passes in order to processed area of 150×50 mm². The processed surface was modified up to 3.5 mm (equal to tool pin height) depth below the surface of plate as shown in Figure 3(a-c). Figure 2(a-b) shows Al-Sc binary phase diagram and optical microstructure.
of cast Al alloy. Sc have low solubility (0.35wt.%) at eutectic point (0.55wt.%) in Al-Sc phase diagram and also consider to have large binding energy. This high energy is able to capture the large amount of vacancies, results are suppression the free migration vacancies to fairly slow diffusion rate. The Al₃Sc particles formed under this condition, normally termed dispersoids which giving strong contribution to the alloy strength. These leads to a good recrystallization resistance and the high temperature stability of the Al alloys. The effectiveness of the dispersoids will depend on the size, spacing and distribution and it can express by Zener drag effect [37]. Figure 2(b) shows optical microstructure of cast Al alloy, which exhibit fine equixed grains (23.0±2.15μm), grain boundary segregations not completely eliminate, porosities, and cast inhomogeneity in matrix. Figure 4 shows optical microstructures after homogenization treatment at different temperatures from 300-500°C at 6h for this alloy. It has to mention that for 6h homogenization time the alloy exhibited optimum hardening effect as shown in Figure 5. After single step homogenization treatment, the non-equilibrium eutectic phases gradually dissolve into the matrix, and then precipitates occur from the matrix during air cooling process. In addition of minor Sc (0.33%) can refine grains, eliminate shrinkages and dendrites, hot tearing for all cast structures at 6h homogenization time. The amount of Sc(0.33%) keep on constant in α-Al region that the expect to single phase homogenized structure
as shown in Al-Sc phase diagram (Figure 2.a). Figure 3(a-c) shows a complete set-up of FSP with the repetitive passes turn to MP-FSP with constant process parameters and conditions as shown in Table 1. It is clear that during processing the materials are transported from advancing side to the retreating side under the rotation of the tool.

Figure 4(a) shows fine grains (24.76±1.94µm) structure with grain boundary segregations, because low temperature (300°C) of non-equilibrium low melting eutectics unable to dissolve completely in matrix. Figure 4(b) shows fine grains (23.50±3.45µm) structure with complete elimination of all types of segregation at 350°C for 6h time, reason for large numbers of Al3Sc particles formation and hardening effects. Figure 4(c) shows fine equiaxed grains (26.27±4.78µm) structure with uniformly distribution and complete elimination of segregation at 400°C for 6h time through out in matrix. It is possibly due to uniformly and homogeneous distribution of hardening particles with slightly higher grain sizes. Figure 4(d) shows finer grains (13.79±2.89µm) structure with more densely distribution rather than any other homogenization temperatures, reason for Al3Sc particles are thermally more stable and numerous generation which provide more grains and precipitation hardening. Some fine cracks are formed may due to thermal stresses at 450°C for 6h. Figure 4(e) shows fine grains (19.43±4.29µm) structure with slightly coarser than 450°C for 6h homogenization treatment, reason for slightly grain coarsening occur for this exothermic reaction, also some hair-line cracks generated due to thermal stresses at 500°C for 6h. Figure 5 shows the Vicker’s hardness vs. time curves at different homogenization temperatures upto 8h of Al alloy.

![Vicker’s hardness curves at different homogenization temperatures for 8h of cast Al alloy.](image)

The Al3(Sc) particles are formed during homogenization treatment and processing has great influence on precipitation behaviour of Al3(Sc) particles. Among the all homogenization temperatures only 400-450°C range exhibited higher hardness values (122-136HV) at 6h time. But homogenization temperature at 500°C exhibited lowest hardness value of 100HV at 6h time. Because minimum...
amount of hardening precipitates formed at this condition. When the time increase to 8h then the hardness value gradually increased to 121HV because formation of fine precipitates and grains refinement are main reason, at the same time some cracks generated due to high temperature distortion or stresses. Figure 6 shows bar diagrams of hardness (HV) and grain size (µm) vs. homogenization treatment at different temperatures for 6h time of Al alloy, in which maximum hardness (122-129HV) reached at 400-500°C range and minimum grain size (13-23µm) obtained at this range. Reasons for maximum numbers of dispersoid distribution of Al3Sc particles of precipitation hardening and anti-recrystallization effects.

Figure 7 shows the FESEM micrograph with EDS analysis of Al alloy, which identified for grain boundary segregation of high impurity contents like Fe (2.29wt.%), Si (2.14wt.%), and Sc (1.07wt.%), respectively. Fe combines with both Al and Si to form secondary intermetallic phases because of low solubility of Fe in Al (max. 0.05% at 650°C), and can form Al3Fe brittle phases. The high content of Si should lead to the formation of Si-inclusions may decrease the electrical conductivity of the Al alloy. The presence of high content impurities affects the nucleation and growth of primary Al3Sc(L12) particles, and thereby enhance the heterogeneous nucleation of α-Al during solidification, and also encouraged a transition on primary Al3Sc phase from a peritectic to eutectic reaction. Figure 8 shows the XRD analysis of Al alloy, which identified several peaks of Sc contents and others like Al3Sc, AlSc, MgZn2, Mg3Zn2, etc. TEM with EDS analysis at different magnifications of cast Al alloy as shown in Figure 9(a-b). The cast alloy has exhibited several nano-sized precipitates (10-50 nm) (indicated by red arrows), specially on the grain boundary regions, and several dislocation-precipitates interactions are observed, and precipitates are mostly inhomogeneous distribution in the matrix. Figure 10 shows optical microstructure of cast 7075 Al alloy after MP-FSP as processing parameters have to be followed in Table 1. The processed zone is exhibiting three distinct zones such as stir zone (SZ) for indication-1, thermomechanically affected zone (TMAZ) for indication-2, and heat affected zone (HAZ) for indication-3 in optical microstructure. In SZ region exhibited most finest microstructures of size 3.17±2.10 µm due to heat input (i.e., ratio of 1000/70 mm per min) of 14.29 impart favourable heat generation during
processing. The sequence of microstructures are analysed by Image J software likely to as-cast average grain size of 23.0±2.15 µm to SZ average grain size of 3.17±2.10 µm, TMAZ average grain size of 7.56±4.67 µm, HAZ average grain size of 12.10±5.39 µm, respectively. This microstructure refined due to stirring action of the tool pin that produces a continuous dynamic recrystallization process [38, 39]. The temperature rise at around 400-500°C and severe plastic deformation during the MP-FSP as a result of new equiaxed grain structure. Another contribution of fine grains achieve in SZ due to formation of nano-sized particles of Al₃Sc, MgZn₂, and η precipitates. It has to mentioned that the 0.2% proof stress, ultimate tensile stress increased with finer grain size, and they obeyed the Hall-Patch relationship: \( \sigma = \sigma_0 + K_d d^{-0.5} \); where \( \sigma_0 \) and \( K_d \) are constants, \( d \) for grain size (µm), \( \sigma \) for 0.2% proof stress (MPa). For example after MP-FSP likely to SZ grain size is 3.17±2.10 µm, \( \sigma = 213.2 \text{ MPa} \) (Table 2), which indicated that the strength increasing with a finer in the grain size. Figure 11(a-c) shows SEM tensile fractographs at different magnifications of cast Al alloy after MP-FSP. At the low magnification, it is clearly indicated black spots (indicated by red arrows) may be due to Zn burning problem encountered for high heat input (14.29) generation at fixed processing parameters. At the medium magnification, it has indicated black spots (indicated by red arrows) with coarse plate shape of Al₃Sc particle or second phase particle exhibit ductile mode of fracture in matrix. At the high magnification, it has indicated several spots (indicated by red arrows) with swallow depth inside some coarse particles may for these second-phase particles agglomeration, its acting for stress concentration centre or crack propagation faster for low fracture strength in the matrix. Table 2 shows mechanical properties of cast Al alloy after MP-FSP, it indicates high tensile strength of 314 MPa and better ductility of 7%, and moderate hardness along the SZ of 114HV due to the elimination of porosity and the breakup of coarse second-phase particles, and homogeneous distribution of Al₃Sc dispersoids and hardening particles [40, 41].

Figure 9: The TEM microstructures with EDX analysis at different magnifications of cast 7075 Al alloy: (a) low magnification (500 nm), (b) high magnification (200 nm).
Figure 10: The optical microstructure after MP-FSP of cast Al alloy. (1000 rpm and 70 mm/min)

Figure 11: SEM tensile fractographs of Al alloy (MP-FSP condition): (a) low magnification (200X), (b) medium magnification (1000X), (c) high magnification (2000X). (1000 rpm and 70 mm/min)

Table 2: Results of mechanical properties have tabulated after MP-FSP of cast 7075 Al alloy.

| 7075 Al alloy | MP-FSP condition | VHN(10kg.) |
|---------------|------------------|------------|
|               | $\sigma_{0.2}$ (MPa) | $\sigma_u$ (MPa) | $\delta$ (%) |
| 213.2         | 314.0            | 7.0        | 114.0        |

4. Conclusions

(1) Significant grain refinement can be achieved in high strength Al-Zn-Mg alloy with the minor addition of scandium (0.33%) during solidification. The Al3Sc particles have high melting point and more stability, making them very ideal nuclei, for refining the as-cast grain size of Al alloy and improving the mechanical properties.

(2) The homogenization treatment of cast Al alloy at different temperatures (300-500°C) have been carried out directly after foundry casting, and it plays a vital role to eliminated microsegregation, hot tearing, and dissolving large soluble non-equilibrium eutectic particles formed during solidification.

(3) For an optimum homogenization condition (450°C for 6h), the most effective dispersoid distribution is obtained by precipitating a large number and uniformly distribute small particles
(MgZn2 and Al3Sc), that is optimized with favour to anti-recrystallization effect, Zener pinning effect and achieved minimum average grain size of 13.79±2.89 µm and hardness of 123HV15kg. respectively.

(4) The MS-FSP makes an effective surface modification of cast alloy. The higher ductility is due to the elimination of porosity and the breakup of coarse second phase particles. The tensile strength after MS-FSP has been improved due to grain refinement and homogenization of precipitate particles of studied aluminium alloy.

(5) The sequence of microstructures are achieved after MP-FSP likely to as-cast average grain size of 23.0±2.15 µm to SZ average grain size of 3.17±2.10µm, TMAZ average grain size of 7.56±4.67 µm, HAZ average grain size of 12.10±5.39 µm. In addition, these microstructures are refined due to stirring action of the tool pin that produces a continuous dynamic recrystallization process for favourable heat input (14.29).

(6) The MS-FSP of cast aluminium alloy had significantly improved mechanical properties of 0.2% proof strength of 213.2 MPa, ultimate tensile strength of 314.0 MPa, ductility of 7.0%, and Vicker's hardness of 114HV, respectively. Also, MP-FSP led to dynamic recrystallization in all conditions to achieve equiaxed grains with high angle grain boundaries (>85%).

References

[1] T. Engdahl, V. Hansen, P.J. Warren, K. Stiller, Mat. Sci. and Eng. A 327, 2002, pp. 59-64.
[2] P.K. Mandal, Mat. Sci. and Met., Vol.4, No.1, 2017, pp. 16-28.
[3] T. Ungar, J. Lendvai, I. Kovacs, J. of Mat. Sci., J.4, 1979, pp. 671-679.
[4] Y. Deng, Z. Yin, K. Zhao, J. Du, Z. He, J. of Alloys and Comp., 530, 2012, pp. 71-80.
[5] P.K. Mandal, Int. J. of Mech. and Prod. Engg. Res. and Dev., Vol. 6, Issue 2, April 2016, pp. 65-76.
[6] Y. Milman, Mat. Sci. Forum, Vols. 519-521, 2006, pp. 339-344.
[7] K.B. Hyde, A.F. Norman, P.B. Prangnell, Acta Materialia, 49, 2001, pp. 1327-1337.
[8] N. Afify, A-F. Gaber, G. Abbady, Mat. Sci. and Applications, 2, 2011, pp. 427-434.
[9] J.H. Li, P. Schumacher, Mat. Sci. and Eng. A, 561, 2013, pp. 78-87.
[10] M.R. Clinch, S.J. Harris, W. Hepples, N.J.H. Holroyd, M.J. Lawday, Mat. Sci. Forum, Vols. 519-521, 2006, pp. 339-344.
[11] Z. Heng-hua, Trans. of Nonferrous Met. Society of China, 18, 2008, pp. 836-841.
[12] M.S. Weglowski, S. Dymek, C.B. Hamilton, Bull. of the Polish Aca. of Sci. Tech. Sci., Vol.61, No.4, 2013, pp. 893-904.
[13] A. Deschamps, Y. Brechet, F. Livet, P. Gomiero, Mat. Sci. Forum, Vols. 217-222, pp. 1281-1286.
[14] X.Z. Li, V. Hansen, J. Gjonnes, J. of Mat. Res., Vol.18, No.8, Aug 2003, pp. 1757-1759.
[15] L.K. Berg, J. Gjonnes, V. Hansen, X.Z. Li, N.J. Ho, J.C. Huang, Mat. Characterization, 61, 2010, pp. 1043-1053.
[16] L-M. Wu, M. Seyring, M. Rettenmayr, W-H. Wang, Mat. Sci. and Engg. A, 527, 2010, pp. 1063-1073.
[17] M. Dixit, R.S. Mishra, K.K. Sankaran, Mat. Sci. and Engg. A, 478, 2008, pp. 163-172.
[18] M. Nishi, K. Matsuda, N. Miura, K. Watanabe, S. Ikeno, T. Yoshiida, S. Murakami, Mat. Sci. Forum, Vols.794-796, 2014, pp. 479-482.
[19] F.A. Costello, J.D. Robson, P.B. Prangnell, Mat. Sci. Forum, Vols. 396-402, 2002, pp. 757-762.