ISSN 0511-5728
The West Indian Journal of Engineering
Vol.34, Nos.1/2, January 2012, pp.80-85

Production and Age-Hardening Behaviour of Borax Premixed SiC reinforced Al-Mg-Si alloy Composites developed by Double Stir-Casting Technique

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(Received 15 June 2011; Revised 30 September 2011; Accepted 13 October 2011)

Abstract: *The production quality and age-hardening behaviour of Al 6063/SiC particulate composites developed using borax additive and a two-step stir casting was investigated. This was aimed at establishing optimum processing and thermal ageing conditions required for the development of Al 6063/SiC$_p$ composites. Al 6063/SiC$_p$ composites containing 6, 9, and 12 volume percent of SiC were produced; and samples representative of each composition were subjected to age-hardening treatment at 180^{o}C, 190^{o}C, and 200^{o}C for holding times ranging from 30 to 180 minutes. Micro-structural characterisation and density measurements were used as a basis of evaluating the porosity and general casting quality of the composites; while hardness measurements were used to study the age hardening behaviour. Experimental results show that Al 6063/SiC$_p$ composites having low porosity levels (≤ 1.6 % porosity) and a good uniform distribution of the SiC particulates in the matrix of the Al 6063 were produced. Compared with the monolithic alloys, the aging response of the 6 and 9 volume percent (vol.%) SiC composites were generally poor, while the 12vol.% SiC composites showed appreciable age-hardening response at temperatures of 180-190^{o}C (at 200^{o}C, the ageing response was poor). Also, the transformation sequence of the composites appeared to be different from that of the monolithic alloy judging from the nature of the hardness profiles.*

Keywords: *Age- hardening; aluminum alloy 6063; stir casting; SiC; Composite; porosity*

1. Introduction

The development of simple, cost effective and technically efficient processing routes for the production of metal matrix composites continues to draw the attention of materials researchers. This quest is becoming more of a research paradigm in a good number of developing countries where there are sustained efforts to extend materials design beyond the use of traditional metals and alloys by the adoption of indigenous technologies (Singla et al., 2009). The attractive properties of MMCs and its higher performance potentials over traditional metals and alloys have given the impetus for many such research efforts (Miracle, 2005; Ray, 1993).

The advantages of metallic composites over their monolithic counterparts include but are not limited to their high specific strength and stiffness, better high temperature performance, low thermal expansion among others (Zhou and Xu, 1997; Hashim et al., 1999). Aluminum based matrix composites remain the most explored metal matrix material for the development of MMCs. This is primarily due to the broad spectrum of properties it offers at low processing cost (Surappa, 2003). They are currently applied in the design of a wide range of components for use in aerospace technology, defence, electronic heat sinks, solar panel substrates, antenna reflectors, automotive drive shaft fins, explosion engine components, sports among others (Surappa, 2003; Srivatsan et al., 2003).

Deriving optimized properties from any selected Al based matrix composite requires a sound knowledge of the material behaviour of the composite which is influenced by such factors as the base Al alloy composition, the manufacturing process, the reactivity between the reinforcement and the matrix, the size, morphology and volume fraction of the reinforcement (Kumar et al., 2010; Christy et al., 2010; Hassan and Aigbodion, 2010).

The current research work is an effort to study the viability of producing Al 6063/SiC$_p$ composites using a two-step stir mixing and borax additive and also, its age hardening behaviour. This processing method is envisaged will reduce drastically the common problem of porosity which is observed in metal matrix composites produced by casting technique.

Several works have been reported on the use of two-step (double) stir casting as a means of improving cast metallic matrix composites (Hassan et al., 2008; Valdez, 2008; Singla et al, 2009). Singla et al (2009) for example compared the production of AMCs using (a.) direct casting (no stirring), (b.) with only manual stirring, and (c.) with two step mixing; and discovered that the two step mixing gives the best uniform distribution of the SiC particulates. They suggested that production of AMCs without the use of two step stirring results in less homogenization of the particulates and higher porosity levels which might be in excess of the acceptable limits. However the process requires the pre-treatment of the SiC particulates in dry oven at 1,100^{o}C to improve wettability between the Aluminum alloy melt and the SiC particulates.

Zhou and Xu (1997) and Kok (2005) have reported that porosity within the range of 2 % and 4 % is acceptable in cast metal matrix composites. The use of borax additive in combination with two-step stirring is to improve wettability without the need to pre-heat the SiC particulates. This procedure has not been reported extensively in literature. The choice of AA 6063 as matrix alloy is because AA 6063 is processed in large quantities at low costs in many developing countries but its potentials for use as Al alloy matrix for composite development have not been explored as extensively as the other age hardenable aluminum alloy series such as the AA 6061, AA 7075, and AA 2024 series (Christy et al., 2010; Gupta and Surappa, 1995; Ehsani and Reihanni, 2004).

2. Materials and Methods

2.1 Materials

The base material for the investigation is wrought aluminum alloy (6063) as received in form of slabs with chemical composition as presented in Table 1. Silicon carbide (SiC) with particle size of 30µm (600grits) was used as reinforcement along with Hydrated sodium tetra borate (borax) $(Na_2B_4)_7.10H_2O$ for improvement of wettability of the molten aluminum alloy 6063 and the silicon carbide particles during melting.

Table 1. Nominal chemical compositions of Al 6063 matrix alloy (wt %)

Matrix alloy	Si	Fe	Cu	Mn	Mg
	0.45	0.22	0.02	0.03	0.50
Matrix alloy	Zn	Ti	Cr	Bal	
	0.02	0.02	0.02	Al	

2.2 Stir Casting

A diesel fired crucible furnace was used for the melting operation. The stir casting set-up for the production of the composites was designed in accordance with Singla et al. (2009). Charge calculations following standard procedures were utilized to estimate the amount of the Al 6063 billets and silicon carbide required to produce 6, 9, and 12 volume percents (vol.%) SiC reinforcements in the composite. The borax which serves as a wetting agent was dehydrated by heating at 250^{o}C for 20 minutes, after which it was mixed with the specified amounts of SiC in ratio 1:2.

The Al 6063 billets were charged into the furnace and melting was allowed to progress until a uniform temperature of 750^{o}C (which is above the liquidus temperature) was attained. The melt was then allowed to cool to 600^{o}C (slightly below the liquidus temperature) to a semi-solid state. At this stage, the silicon carbide and dehydrated borax mixture was added into the melt and manual stirring of the slurry was performed for 20 minutes. An external temperature probe was utilized in all cases to monitor the temperature readings of the furnace.

After the manual stirring, the composite slurry was reheated and maintained at a temperature of 750^{o}C +-10^{o}C (above the liquidus temperature) and then mechanical stirring was performed. The stirring operation was performed for 10 minutes at an average stirring rate of 400rpm. Casting was then performed on prepared sand moulds at a pouring temperature of 720^{o}C.

2.3 Age-Hardening Treatment

Age-hardening of selected samples from each volume percent of the composites produced was performed. The selected samples were solution-treated in the furnace at 560^{o}C for 2 hours, followed by water quenching. Thereafter, ageing was performed at temperatures of 180^{o}C, 190^{o}C, and 200^{o}C for holding periods ranging 30 minutes to 360 minutes followed by water quenching.

2.4 Hardness Measurement

Hardness tests were performed using a Vickers Hardness Tester (LECO AT 700 Microhardness Tester). A direct load of 50gf (490 mN) was applied on flat smoothly polished specimens of the composites for 10 seconds and the hardness readings evaluated following standard procedures. Multiple hardness tests were performed on each sample and the average value taken as a measure of the hardness of the specimen.

2.5 Density Measurement

The density measurements were carried out to determine the porosity levels of the samples. This was achieved by comparing the experimental and theoretical densities of each volume percent SiC reinforced composite. The experimental density of the samples was evaluated by weighing the test samples using a high precision electronic weighing balance with a tolerance of 0.1mg. The measured weight in each case was divided by the volume of respective samples. The theoretical density was evaluated by using the rule of mixtures given by:

$$\rho_{Al\ 6063/SiCp} = Vol_{Al} \times \rho_{Al\ 6063} + Vol_{SiC} \times \rho_{SiC} \quad \text{.......... Eq.1}$$

Where, $\rho_{Al\ 6063/SiCp}$ = Density of Composite, Vol_{Al} = Volume fraction of AA 6063, $\rho_{Al\ 6063}$ = Density of Al 6063, Vol_{SiC} = Volume fraction SiC, and ρ_{SiC} = Density of SiC.

The percent porosity of the composites was evaluated using the relations:

$$\%\ porosity = \{(\rho_T - \rho_{EX}) + \rho_T\} \times 100\% \quad \text{........ Eq.2}$$

Where, ρ_T = Theoretical Density (g/cm^3), ρ_{EX} = Experimental Density (g/cm^3)

2.6 Microstructure

The microstructural investigation was performed using a Datteng Software-Driven Metallurgical Microscope. The specimens for the optical microscopy were polished using a series of emery papers of grit sizes ranging from 500µm-1,500µm; while fine polishing was performed using polycrystalline diamond suspension of particle sizes ranging from 10µm-0.5µm with ethanol solvent. The specimens were etched with 0.5% HF solution by swabbing for 3-6 minutes, followed by rinsing in water and drying before observation in the optical microscope.

3. Results and Discussion

3.1 Production Quality

1) *Microstructure* - The microstructure of the composites can be used as an important indicator of the quality of the composites and a measure of the effectiveness of the technique adopted for the production. Figures 1, 2 and 3 show three representative optical micrographs for the SiC reinforced Al 6063 composites produced. It is observed that the distribution of the SiC particulates in the matrix of the Al 6063 is fairly uniform with minimal particle clusters. This indicates that the technique utilized for the production of the composite is efficient.

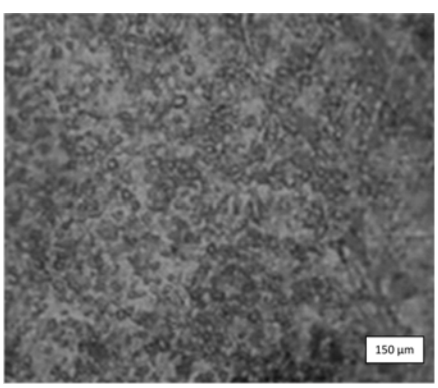

Figure 1. Optical micrograph of Al 6063/ 6SiC$_p$ composite

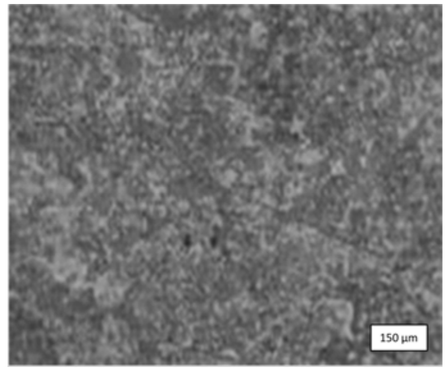

Figure 2. Optical micrograph of Al 6063/ 9SiC$_p$ composite

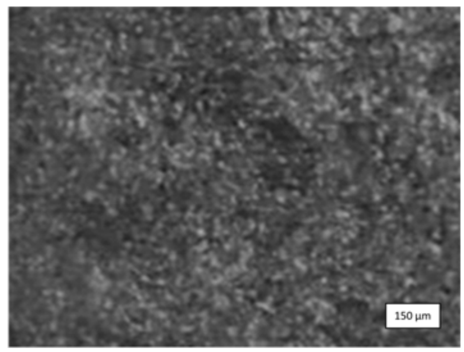

Figure 3. Optical micrograph of Al 6063/ 12SiC$_p$ composite

The achievement of uniform dispersion of SiC particles in the microstructure of Al based matrix composites has been used as a strong measure of the quality of composites (Eshani and Reihani, 2004).

2) *Percent Porosity* - The percent porosity, and its size and distribution in cast metal matrix composites play an important role in controlling the mechanical properties. It is thus necessary that porosity levels be kept to a minimum if the desired high performance in service applications would be achieved. In this respect, Table 2 presents the comparison of the theoretical densities and the experimental density values of the Al 6063/SiCp composites produced, which were utilized to determine the porosity levels in the composites.

Table 2: Comparison of theoretical and experimental densities of Al 6063/SiC$_p$ composites

Material	Density (g/cc)	Weight % of Reinforcement			
		0	6	9	12
Al 6063/ SiC$_p$	Theoretical Density	2.70	2.724	2.736	2.748
	Experimental Density	2.64	2.68	2.69	2.72
	Percentage Porosity, %	2.2	1.6	1.54	1.178

It was observed that for all SiC volume percent reinforced Al 6063 composites produced, the theoretical and experimental densities were very close as reflected by the maximum of 1.6 % porosity obtained. Also, it is observed that the porosity in the monolithic alloy cast without stirring had a slightly higher porosity level in comparison with the composites produced by the two step stirring and Borax additive process.

Many researchers (e.g., Kok, 2005; Prabu et al., 2006; Singla et al., 2009) have used alternative stirring processes and reported that porosity levels were within the range of 2-4%, which were referred to as acceptable levels of porosity in cast composites. This indicates that the processing technique adopted in this research is highly efficient for the production of Al 6063/SiC particulate composites. Porosity in composites results primarily from air bubbles entering the slurry during the stirring period or as air envelopes to the reinforcing particles (Ray, 1993; Pakdel et al., 2007).

The adoption of the two-stage stirring and the addition of borax are responsible for the low porosity levels and the minimal casting defects in the composites produced. The first stirring of the preheated borax / SiC particles and the molten Al 6063 slurry was performed in the semi-solid state to prevent the relatively denser SiC particles from floating on the surface of the melt due to high surface tension and poor wetting between the particles and the melt (Singla et al., 2009).

In the semi-solid state, there is an increase in the viscosity of the melt which helps prevent the floating of the SiC particles. Singla et al (2009) reported that the agitations induced in the slurry during the manual stirring operation aids in breaking the surface gas layers surrounding the particles as well as spread the liquid metal onto the surfaces of the particles, thereby helping to improve wettability. Mechanical stirring is then performed. This second step reheats the slurry above the liquidus temperature and stirs at this temperature so as to improve the particle distribution. This improves the fluidity of the melt and aids improved particle dispersion which reduces the effects of particle sedimentation.

The mixing of borax with the SiC particles aids proper wetting of the SiC in the Al 6063 matrix by enhancing particulate/ matrix interfacial bonding and improved particle distribution which reduces the chances of the development of casting defects (Cameron et al., 1987). Figures 4 and 5 show sample castings from the mould immediately after shakeout, and the composite bars after fetlling operation, respectively. Figure 6 shows some cut samples from the composites for hardness test.

Observations from visual examination and machining confirm that there were minimal casting defects on the composites produced. The densities of the composites are higher than that of the monolithic alloy, and also the density increases with increase in the volume fraction of SiC is expected.

Figure 4. Castings from the mould immediately after shakeout

Figure 5. The composite bars after fetlling operation

Figure 6. Cut samples from the composites for hardness test

3.2 Age-Hardening Behaviour of the Composites

The ageing responses of the Al 6063/SiC_p composites at varying temperatures of 180^{o}C, 190^{o}C and 200^{o}C were studied. The preliminary observations from the assessment of the hardness results indicated that the hardness of the composites increased with increase in volume percent of the SiC particulate (see Figures 7, 8 and 9, respectively). It was noticed that in comparison with the monolithic alloys, the aging response of the 6 and 9 vol.% SiC composites were generally poor as no increase in hardness was obtained for both volume percents after ageing treatment, while the 12 vol.% SiC composites showed appreciable age-hardening response at temperatures of 180^o-190^{o}C as considerable increases in hardness were observed for both temperatures (at 200^{o}C its ageing response was poor). This suggests that the ageing behaviour of the Al 6063/SiC_p composites is influenced by the volume percent of SiC and the ageing temperature.

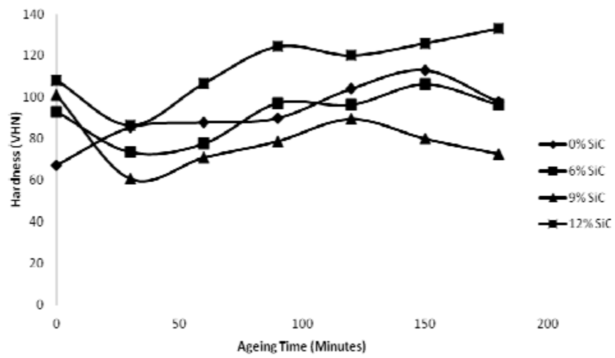

Figure 7. Thermal Ageing Curves for the Al 6063/SiC_p composites at 180^{o}C

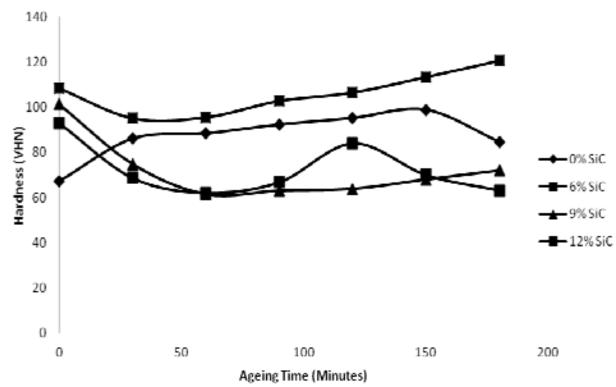

Figure 8. Thermal Ageing Curves for the Al 6063/SiC_p composites at 190^{o}C

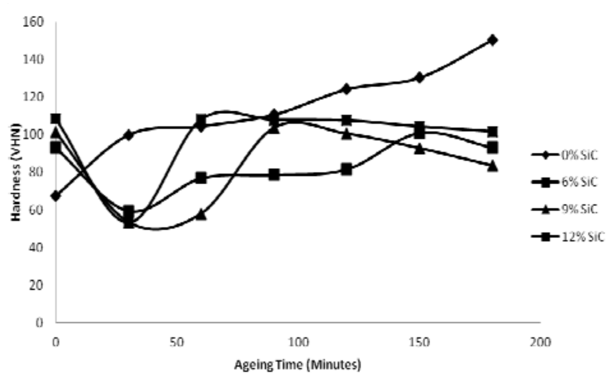

Figure 9. Thermal Ageing Curves for the Al 6063/SiC_p composites at 200^{o}C

Khalifa and Mahmoud (2009) reported that increased SiC content leads to increased acceleration of the aging kinetics and increased hardness. The transformation sequence of the composites appears to be different from that of the monolithic alloy judging from the nature of the hardness profiles. It is noticed that for all the ageing temperatures, there is an initial drop in hardness for the composites during age hardening treatment. This is in contrast to the behaviour of the monolithic alloy which is observed to exhibit a progressive increase in hardness until peak hardness is attained. This phenomenon (initial drop in hardness) could be attributed to stress relief (recovery) of the composites which preceded the precipitation reaction. Dislocations are well-known to be created in metal matrix composites as a result of the thermal mismatch between the metal matrix and the reinforcing material during cooling (Ehsani and Reihani, 2004).

It has been observed that for structures subjected to thermal treatments at temperatures where precipitation and recrystallization (recovery) are thermodynamically feasible, the phase reaction with the lower activation energy will most likely precede in the transformation process (Alaneme and Kamma, 2010; Martin et al., 1997). Thus it is the opinion of the authors that the initial softening observed is most likely due to recovery process (to stress relief the composites).

5. Conclusions

The use of borax additive and two-step stir casting technique resulted in the production of Al 6063/SiC_p composites having low porosity level (≤ 1.6 % porosity) and a good uniform distribution of the SiC particulates with minimal particle clusters in the matrix of the Al 6063. Compared with the monolithic alloys, the aging response of the 6 and 9 vol.% SiC composites was generally poor, while the 12vol.% SiC composites showed appreciable age-hardening response at temperatures of 180^o-190^{o}C as considerable increases in hardness were observed for both temperatures (at 200^{o}C its ageing response was poor).

Moreover, the transformation sequence of the composites appears to be different from that of the monolithic alloy judging from the nature of the hardness profiles. For the composites, there is an initial drop in hardness during age hardening treatment while the monolithic alloy in contrast exhibits a progressive increase in hardness until peak hardness is attained.

References:

Alaneme, K.K. and Kamma, C.M. (2010), "Phase transformation studies of a low alloy steel in the (α + γ) phase region", *Materials Research*, Vol.13, No.1, pp.119-123

Cameron, T.B., Swanson, W.W., Tartaglia, J.M. and Cox, T.B. (1987), *Production of Aluminium – SiC Composite using Sodium Tetraborate as an Addition Agent*, U.S Patent 4,667,008, available at: freepatentonline.

Christy, T.V., Murugan, N. and Kumar, S. (2010), "A comparative study on the microstructures and mechanical properties of Al 6061 alloy and the MMC Al 6061/TiB2/12p", *Journal of Minerals and Materials Characterization and Engineering*, Vol.9, No.1, pp.57-65

Ehsani, R. and Seyed Reihani, S.M. (2004), "Aging behaviour and tensile properties of squeeze cast Al 6061/ SiC metal matrix composites", *Scientia Iranica*, Vol. 11, No 4, pp.392-397

Gupta, M. and Surappa, M.K. (1995), "Effect of weight percentage of SiC particles on the ageing behaviour of Al 6061/ SiC metal matrix composites", *Journal of Materials Science Letter*, Vol.14, pp.283

Hashim, J., Looney, L. and Hashim, M.S.J. (1999), "Metal matrix composites production by the stir-casting method", *Journal of Materials Processing Technology*, Vol.92-93, pp.1-7

Hassan, S.B., Aponbiede, O. and Aigbodion, V.S. (2008), "Precipitation hardening characteristics of Al-Si-Fe/SiC particulate composites", *Journal of alloys and Compounds*, Vol.466, Nos. 1-2, pp.268-272

Hassan, S.B. and Aigbodion, V.S. (2010), "The study of the microstructure and interfacial reaction of Al-Cu-Mg/Bagasse ash particulate composite", *Journal of Alloy and Compounds*, Vol.491, No.1, pp.571-574

Khalifa, T.A. and Mahmoud, T.S. (2009), "Elevated temperature mechanical properties of Al alloy AA6063/SiCp MMCs", *Proceedings of the World Congress on Engineering 2009*, Vol.11, July 1-3.

Kok, M. (2005), "Production and mechanical properties of Al2O3 particle reinforced 2024 aluminum alloy composite", *Materials Processing Technology*, Vol.161, pp. 381-387

Martin, J.W., Doherty R.D. and Cantor B. (1997), *Stability of Microstructure in Metallic Systems*, 2nd Edition, Cambridge University Press, United Kingdom

Miracle, D.B. (2005), "Metal matrix composites - from science to technological significance", *Composites Science and Technology*, Vol. 65, No.15-16, pp 2526-2540

Pakdel, A., Farhangi, H. and Emamy, M. (2007), "Effect of extrusion process on ductility and fracture behaviour of SiCp/ aluminum-alloy composites", *Proceedings of 8th International Fracture Conference*, Istanbul, Turkey, November, pp.460-470

Prabu, S.B., Karanamoorty, L., Kathiresan, S. and Mohan, B. (2006), "Influence of stirring speed and stirring time on distribution of particulates in cast metal matrix composite", *Journal of Materials Processing Technology*, Vol.171, No.2, pp.268-273

Ray, S. (1993), "Review: synthesis of cast metal matrix particulate composites", *Journal of Materials Science*, Vol.28, pp.5397-5413

Singla, M., Dwivedi, D.D., Singh, L. and Chawla, V. (2009), "Development of aluminum based silicon carbide particulate metal matrix composites", *Journal of Minerals and Materials Characterization and Engineering*, Vol.8, No.6, pp.455-467

Srivatsan, T.S., Al-Hajri Meslet, Smith, C. and Petraroli, M. (2003), "The tensile response and fracture behavior of 2009 aluminum alloy metal matrix composite", *Materials Science and Engineering A*, Vol. 346, pp 91-100

Surappa, M.K. (2003), "Aluminum matrix composites: challenges and opportunities", *Sadhana*, Vol.28, Parts 1&2, pp.319-334

Valdez, S., Campillo, B., Perez, R., Martinez, L. and Garcia, H. (2008), "Synthesis and microstructural characterisation of Al-Mg alloy-SiC particulate composite", *Materials Letters*, Vol.62, pp.2623-2625

Veeresh Kumar, G.B., Rao, C.S.P., Selvaraj, N. and Bhagyashekar, M.S. (2010), "Studies on Al (6061) and 7075-Al203 Metal Matrix Composites", *Journal of Minerals & Materials Characterization and Engineering*, Vol. 9, No.1, pp.43-55

Zhou, W. and Xu, Z.M. (1997), "Casting of SiC reinforced metal matrix composites", *Journal of Materials Processing Technology*, Vol.63, pp.358-363

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