Effect of Transition Metals on Thermal Stability and Mechanical Properties of Aluminum

Aamir Khan, Muneer Baig, and Abdulhakim AlMajid

Abstract—**In the present study, binary alloys Al-X (5wt%) where (X =Cr,Ti) and a ternary alloy Al-5Cr-5Ti (all in wt.% unless mentioned) were processed via mechanical alloying technique to investigate its thermal stability and mechanical properties at high temperatures. The powders were mechanically alloyed in a ball mill for 100hrs with a ball to powder ratio of 10:1. The bulk samples of these powders were obtained by sintering in a high frequency induction sintering furnace. Change in the phases and crystallite size was evaluated by X-ray diffractometer. Crystallite size of sintered samples for binary composition Al-5Cr and Al-5Ti was estimated to be 44 nm and 46 nm respectively, whereas for ternary alloy it was noted as 42 nm. The compressive yield strength of Al-5Ti, Al-5Cr and Al-5Ti-5Cr at room temperature was found to be 471MPa, 517MPa and 545MPa respectively. Vickers micro hardness for Al-5Cr, Al-5Ti and Al-5Cr-5Ti at room temperature was found to be 146Hv, 130Hv and 160Hv. The ternary alloy, Al-5Ti-5Cr exhibited superior mechanical and microstructural properties both at ambient and elevated temperatures. The establishment of intermetallic precipitates plausibly played a vital role in enhancement of strength and hardness at higher temperatures.**

Index Terms—**Aluminum alloys, ball milling, transition metals, thermal stability.**

I. INTRODUCTION

In the last decade constant efforts have been made to develop light weight high strength alloys due to their vast applications in automobile and aerospace industry [1], [2]. Aluminum alloys belong to a class of engineering materials that exhibit good mechanical properties and possess a high strength to weight ratio. However, the application of these alloys is limited to the manufacturing of components that are subjected to low temperatures. This is because, the thermal stability of alloys is relatively lower from other engineering materials [3]. Decrease in strength of Al-based alloys with increase in temperature is dedicated to grain growth [3]. Controlling the grain growth with increase in temperature has become a challenging task. Thus, a procedure to stable the grain size and increase the strength at elevated temperatures has become much more important.

The subject of inter related terms grain growth and thermal stability of nanostructured aluminum alloys has accelerated many researchers to expand their area of significant work on this particular area. Several studies have been performed on thermal stability of nanostructured aluminum alloys [4]-[6]. Generally, kinetic stabilization mechanism and thermodynamic stabilization mechanism are two basic mechanisms to control the grain growth at higher temperatures. In kinetic stabilization mechanism grain movement is hindered by solute drag, precipitation or dispersion of second phase particles, whereas in thermodynamic stabilization mechanism the grain growth is controlled by reducing the excessive grain boundary energy almost to zero by solute segregation at the grain boundary. Thus, there is no driving force available for further grain coarsening [7], [8]. On other hand aluminum matrix reinforced with transition metals had shown a significant increase in thermal stability due to their lower diffusivity. The main cause for the increase in strength with the addition of transition elements is dedicated to finely dispersed microstructure, formation of metastable intermetallic phases and high super saturation of solid solutions [9]. Among all of the transition metals Cr and Ti is found to increase the thermal stability in aluminum. Apart from this, the solubility limit of Cr and Ti in fcc Al are found to be(> 0.01 at%,>0.2at%) [10]. One of the most efficient techniques to increase the solid solubility and increase solute content in solid solution is Mechanical Alloying (MA). MA does not only establish supersaturated solutions easily but it also refines the size of powder particles to nanometer range which improves the mechanical properties [11]. However, based on the investigations there is a need to develop a thermally stable aluminum composite alloy for high temperature applications.

In the present study, the as milled Pure Al and Al-TM (Transition metals) are fabricated by mechanical alloying technique. Secondly the thermal stability of these processed alloys has been studied at elevated temperatures by means of compression testing and Vickers Micro Hardness.

II. EXPERIMENTAL PROCEDURE

Elemental powders Al (99.9% purity), Ti and Cr (Alfa Aesar) were weighted with the required proportions and loaded in a stainless steel vials containing steel balls. In order to avoid contamination of the powders, the vial is sealed in a glove box maintained under vacuum. Ball to powder ratio was maintained as 10:1 throughout the milling. The MA of powders was carried out in a Fritsch pulverisette P5 planetary ball mill at a speed of 200rpm for 100hrs.The milling operation was programmed such that, there is 15 minutes of pause after every 30 minutes of milling. It was well known repeated fracturing and re-welding occurs during milling. Therefore, to inhibit the agglomeration in prolonged milling (100 hours) 1wt% Stearic acid was used as a process control agent. After milling, the powder was transferred into a

Manuscript received June 15, 2018; revised August 23, 2018.

Aamir Khan is with King Saud University, Riyadh, KSA (e-mail: aamirk930@gmail.com).

Muneer Baig is with Prince Sultan University, Riyadh, KSA (e-mail: mbaig@psu.edu.sa).

Abdulhakim Almajid is with Prince Sultan University and King Saud University, Riyadh, KSA (e-mail: aalmajid@ksu.edu.sa).

doi: 10.18178/ijmmm.2018.6.6.409

cylindrically graphite die with 10mm in diameter and was subjected to sintering operation in a high frequency induction heating furnace at 50 MPa of load and 550 degree Celsius for 6 minutes.

A. Characterization and Microstructure

The characterization of powder and sintered samples was achieved on Shimadzu X-Ray Diffractometer using Cu-kα radiation (λ=0.154 nm) at a scan rate of 1 º/min for a 2θ value of 30 to 80 degrees. In case of sintered samples, the samples were thoroughly polished to remove the surface impurities, with different ranges of sand paper starting with coarse and ending with fine grit size paper. The average crystallite size of the milled powders and sintered samples was calculated by Debye Scherer equation from the first two peaks of aluminum. JEOL model JSM-6610LV FESEM with energy dispersive X-Ray analyzer was used to study the microstructural properties of the milled powders with different compositions. Moreover, EDX analysis from the FESEM confirmed the appropriate mixing of the powder in every composition.

B. High Temperature Compression Testing and Vickers Micro Hardness

Instron model no.3385H attached with the external furnace was used to carry out the compression testing at a strain rate of $10^{-2}s^{-1}$. Compression testing of the sintered samples was performed at the room temperature and elevated temperatures 300,400 and 550 ℃ respectively. Friction plays an important role during compression so in order to avoid this effect; the samples were well lubricated with high temperature grease on both top and bottom surfaces of the sample. For elevated temperatures testing, an external thermometer was attached to confirm the temperature of the specimen. Once the desired temperature has been reached the samples were further held for 30 minutes to assure the uniform temperature distribution. The surface of the samples was mirror polished and measurements of micro hardness were performed on Zwick-Roell micro hardness tester. The parameters for the micro hardness was carried out at load of 200gf with dwell time of 10s and an average of 8 readings were noted. To study the hardness behavior of alloys at elevated temperatures the samples were further heat treated at 300,400 and 550 degrees for 30 minutes followed by quenching in water.

III. Results and Discussions

Fig. 1 shows the image of SEM-EDX analysis for Al-5Ti-5Cr 100 hours milled powder which gives the outcome that Ti and Cr powders were well mixed in the aluminum. Moreover, EDX analysis revealed that the amount of weight percentages in selected area were 90 wt.% of Al,4.99 wt.% of Ti and 4.63 wt.% of Cr and the remaining quantity was found to be carbon from the tape which is very close near to the added proportions. It can also be noticed that there is an evidence of agglomeration from large particles due to prolonged milling of 100 hours which clearly indicates that 1 wt.% of stearic acid was insufficient to inhibit the agglomeration [12].

Fig. 2 shows the image of XRD pattern of 100 hours milled powder with different compositions. From the image it is marked that there are no any intermetallic phases formed after milling but shift in the peak of Al (111) was recognized. The enlarged view of XRD pattern demonstrates with reference to pure as milled Al peak there exists the shift of peaks for different composition milled powders towards the lower angle of 2θ position. The shift in the angle towards the lower angle position is referred to increase in the lattice parameter which explains the formation of solid solutions in Al lattices [13]. Table I represents the crystallite size and lattice parameter for different composition milled powders. It can be seen with increase in alloying element that is in Al-5Ti-5Cr compared from binary composition the grain refinement was acquired due to which the crystallite size decreased and the lattice parameter increased which is in full promise of the formation of solid solutions in Al lattice. Apart from this intensity decreased with increase in alloying element which is also another agreement for the grain refinement.

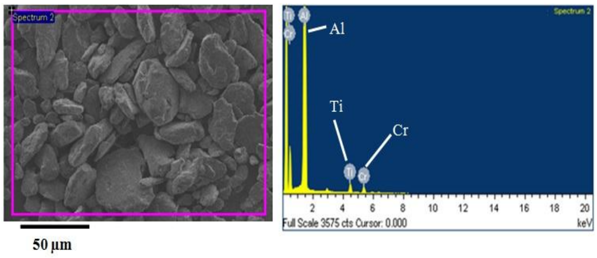

Fig. 1. SEM-EDX Image of Al-5Ti-5Cr 100 hours milled powder.

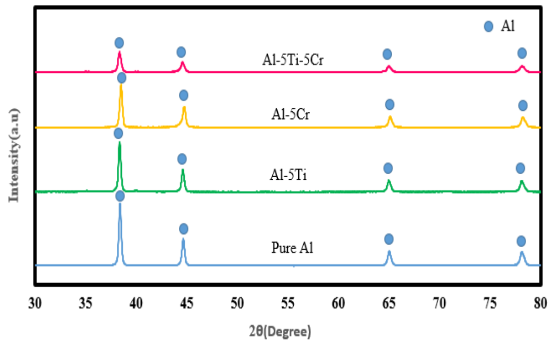

Fig. 2. X-Ray diffraction pattern of 100 hours milled powders.

TABLE I: Crystallite Size and Lattice Parameter of 100 Hours Milled Powders

Milled Powders	Crystallite Size (nm)	Lattice parameter (nm)
Pure Al	48	0.4046
Al-5Ti	33.727	0.40593
Al-5Cr	32.1	0.40598
Al-5Ti-5Cr	24.577	0.40605

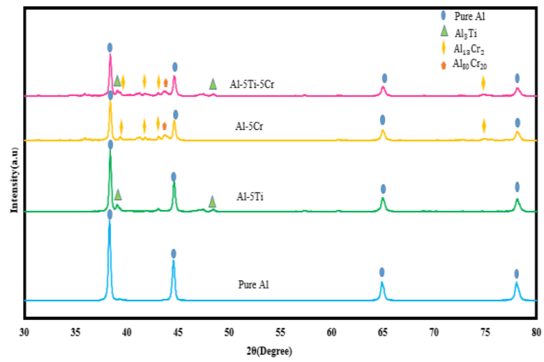

Fig. 3. X-Ray diffraction pattern of high frequency sintered alloys.

Fig. 3 shows the XRD pattern of high frequency induction sintered samples with different compositions which was much sufficient to develop the intermetallic phases. These intermetallic precipitates formed played a vital role in strengthening the mechanical properties at elevated temperatures. On other hand the crystallite size of sintered specimens Al-5Ti, Al-5Cr, Al-5Ti-5Cr was found to be 46nm, 44nm and 42 nm respectively. The small change in the crystallite (grain size) when compared to the powders from Table I is credited to the rapid solidification that is 550 ℃ temperature at load of 50 MPa for 6 minutes.

A. Thermal stability of Sintered Alloys

The thermal stability of sintered alloys is evaluated by Vickers micro hardness and high temperature compression testing. The sintered samples with different compositions were further heat treated in vacuum furnace by batches at different temperatures 300,400 and 550 °C and was quenched in water to evaluate its thermal stability. Fig. 4 represents the hardness data at different temperatures followed by subsequent quenching. Hardness for Pure Al dropped with increase in temperature but for remaining Al-TM alloys were found to be thermally stable at 300 degrees. At 400 ℃ hardness for alloys were dropped and at 550 ℃ the hardness was again increased. This demonstrated that at 400 ℃ there was formation of metastable phases which lead drop in hardness but at the temperature of 550 ℃ this alloys formed a stable intermetallic phases which further increased their hardness. Zawrah *et al.* [13] stated in Al-Fe-Ti-Cr alloy that the formation of metastable phases for Cr and Ti is between 330 ℃ and 450 ℃ and at 550 °C occurrence of the stable precipitates takes place. From this it's in clear agreement that due to the formation of unstable phases at 400 ℃ the hardness was found to be dropped in all Al-TM alloys and at 550 ℃ the formation of stable phases lead to increase in the hardness.

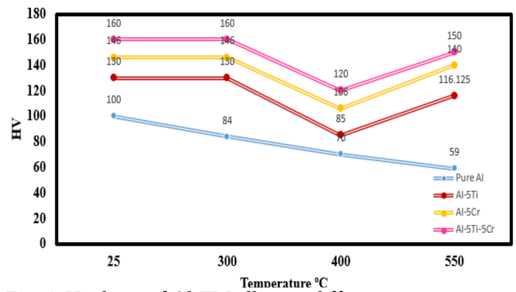

Fig. 4. Hardness of Al-TM alloys at different temperatures.

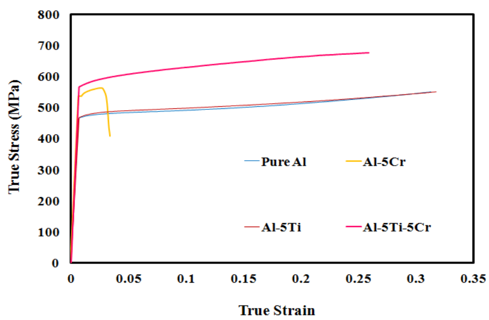

Fig. 5. Room temperature true stres-strain compression behavior of Al-TM alloys.

Fig. 5 represents the compression true stress-strain behavior of pure Al, Al-5Ti, Al-5Cr and Al-5Ti-5Cr alloys at room temperature. The yield strength for the alloys was found to be 468, 471,517and 545MPa, respectively. These results clearly demonstrate that with an increase in alloying elements the strength of aluminum based alloy improved. Furthermore, the formation of secondary phase or intermetallic particles during the milling and sintering process is attributed to the increase in strength of the alloys. However, it was noted that with the increase in strength the ductility of the Al-5Cr alloy decreased.

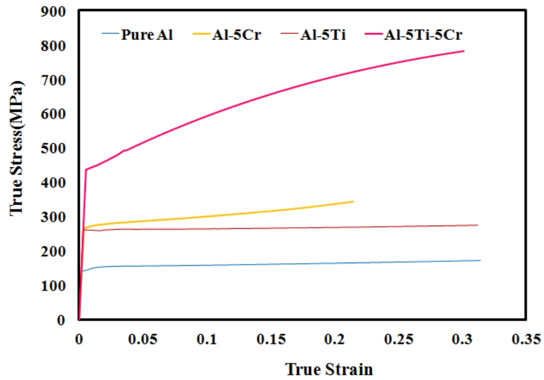

Fig. 6. At 300 ℃ true Stres-strain compression behavior of Al-TM alloys.

Fig. 6 displays the true stress-strain behavior of pure Al, Al-5Ti, Al-5Cr and Al-5Ti-5Cr alloys at 300 ℃. Yield strength of the alloys was found to be 155.3, 257, 263.5 and 439.37 MPa. In fact, the Al-5Ti-5Cr alloys showed the high strength of 439.37 MPa which could be dedicated to the intermetallic phases formed. It is further observed that the strength of the ternary alloy showed higher strength compared to the binary alloy system. The ternary alloy possessed higher concentration of the alloying elements that resulted in formation of higher percentage of precipitates in the alloy compared to binary alloy system. It was also observed that, with an increase in temperature (during high temperature experiments) the ductility of the alloys increased.

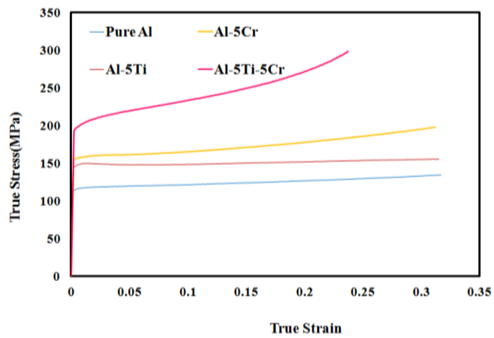

Fig. 7. At 400 ℃ true Stres-strain compression behavior of Al-TM alloys.

To investigate the strength of the alloys at elevated temperatures, compression experiments were performed at higher temperatures. Fig. 7 represents the true stress-strain behavior of pure Al, Al-5Ti, Al-5Cr and Al-5Ti-5Cr alloys at 400 ℃. Yield strength of the alloys was found to be 109,147.25,158 and MPa and 194.52 MPa, respectively. As expected, increase in the deformation temperature lead to decrease in the yield strength of all the alloys that were investigated. However, Al-5Ti-5Cr alloy showed superior properties and was attributed to the increase in the concentration of the intermetallic precipitates when compared to the other alloys investigated.

Fig. 8 displays the true stress the true stress-strain behavior of pure Al, Al-5Ti, Al-5Cr and Al-5Ti-5Cr alloys at 550 ℃. The yield strength of these alloys were found to be 52, 62,65 and 114.54 MPa. Furthermore, increase in temperature resulted in drop of the yield strength which could be related to the grain coarsening of the intermetallic precipitates. However, this confirmed that the increase in the alloying element in aluminum has larger strengthening effect.

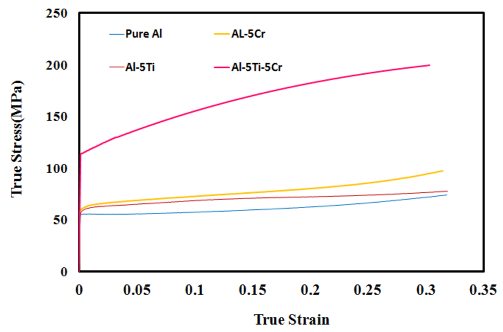

Fig. 8. At 550 ℃ true Stres-strain compression behavior of Al-TM alloys.

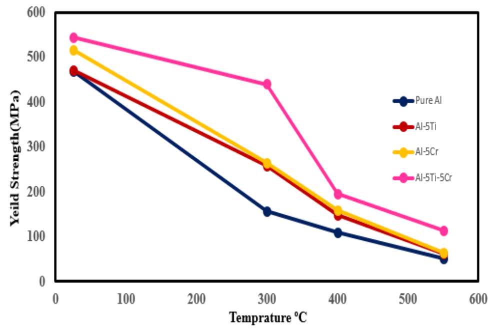

Fig. 9. Yield Strength responses of Al-TM alloys at different temperatures.

Fig. 9 displays the yield strength responses with the increase in temperature which demonstrates that transition metals played an important role in increasing the strength of the alloys at higher temperatures. It was also observed that in binary alloy system Cr was found to be a better stabilizing element at elevated temperatures compared to the titanium. This could be due to the early formation of thermally stable precipitates in Al-Cr alloys when compared to Al-Ti alloys.

IV. Conclusion

Al-TM Nanocrystalline alloys were successfully fabricated by sintering technique of mechanical alloyed powders for high temperature applications. The microstructural and thermal stability of the alloys were studied and briefly explained as follows.

1) In binary alloy composition Al-5Cr and Al-5Ti, the Al-5Cr showed the superior properties compared to the Al-5Ti alloys. This demonstrated that Cr had a better strengthening effect in aluminum at high temperatures when compared to the titanium.
2) Al-5Ti-5Cr alloy compared to other binary compositions exhibited better properties in terms of lowest crystallite size, high strength and improved hardness which are dedicated to the increase in alloying element that leads in the grain refinement and formation of more intermetallic precipitates.
3) The intermetallic precipitates formed probably played an important role in enhancement of strength compared to pure aluminum.

References

[1] S. Pedrazzini *et al*., "Strengthening mechanisms in an Al-Fe-Cr-Ti nano-quasicrystalline alloy and composites," *Materials Science and Engineering: A*, 672: pp. 175-183, 2016.
[2] M. Rahimian *et al.*, "Microstructural Transition and Elevated Temperature Tensile Properties of Modified Al–Si–Cu–Mg Alloy," *Light Metals*, Springer, pp. 419-425, 2017.
[3] A. Prosviryakov and K. Shcherbachev, "Strengthening of mechanically alloyed Al-based alloy with high Zr content," *Materials Science and Engineering: A*, vol. 713, pp. 174-179, 2018.
[4] E. Balducci *et al.*, "Thermal stability of the lightweight 2099 Al-Cu-Li alloy: Tensile tests and microstructural investigations after overaging," *Materials & Design*, vol. 119, pp. 54-64, 2017.
[5] C. Suwanpreecha *et al.*, "New generation of eutectic Al-Ni casting alloys for elevated temperature services," *Materials Science and Engineering: A*, vol. 709, pp. 46-54, 2018.
[6] F. Průša, V. Kučera, and D. Vojtěch, "An Al-17Fe alloy with high ductility and excellent thermal stability," *Materials & Design*, vol. 132, pp. 459-466, 2017.
[7] X. Cai *et al*., "A bulk nanocrystalline Mg–Ti alloy with high thermal stability and strength," *Materials Letters*, vol. 210, pp. 121-123, 2018.
[8] V. S. Muthaiah and S. Mula, "Effect of zirconium on thermal stability of nanocrystalline aluminium alloy prepared by mechanical alloying," *Journal of Alloys and Compounds*, vol. 688, pp. 571-580, 2016.
[9] K. Saksl, D. Vojtěch, and J. Ďurišin, "In situ XRD studies on Al–Ni and Al–Ni–Sr alloys prepared by rapid solidification," *Journal of Alloys and Compounds*, vol. 464, no. 1, pp. 95-100, 2008.
[10] M. Zawrah and L. Shaw, "Microstructure and hardness of nanostructured Al–Fe–Cr–Ti alloys through mechanical alloying," *Materials Science and Engineering: A*, vol. 355, no. 1, pp. 37-49, 2003.
[11] C. Suryanarayana, "Mechanical alloying and milling," *Progress in Materials Science*, vol. 46, no. 1, pp. 1-184, 2001.
[12] M. Baig, H. R. Ammar, and A. H. Seikh, "Thermo-mechanical responses of nanocrystalline Al–Fe alloy processed using mechanical alloying and high frequency heat induction sintering," *Materials Science and Engineering: A*, vol. 655, pp. 132-141, 2016.
[13] C. Suryanarayana and M. G. Norton, "X-ray diffraction: A practical approach," New York: Plenum Press, 1998.

Aamir Khan received his bachelor's degree in mechanical engineering from Shadan College of Engineering and Technology, Jawaharlal Nehru Technological University Hyderabad, India in 2012. He joined as a master's student in Mechanical Engineering Department at King Saud university in the year 2014.

Muneer Baig received the B.S. degree in mechanical engineering from Osmania University, India in 1998, and the M.S. degrees in mechanical engineering from University of New Orleans, USA 2000. He received his Ph.D in mechanical engineering from University of Maryland Baltimore County. He Joined King Saud University as an assistant professor from 2010-2017. He is currently employed in Prince Sultan University as an assistant professor.

His professional interests include high strain rate deformation behavior of materials.

He has 2 patents and several publications in peer reviewed journals.

Abdulhakim Almajid received his B.S. degree from King Saud University in 1992 in Electrical Engineering, M.S. degree in 1995 from Washington University in St. Louis, USA, in Mechanical Engineering, and Ph.D degree in 2002 from University of Washington in Seattle in Mechanical Engineering. He has joined Kind Saud University in 2002 as an assistant professor, He became the Chairman of the Mechanical Engineering Department in KSU from 2005 to 2011. He also became the technical director of the Center of Excellence for Research for Engineering Materials (CEREM) in KSU from 2008 to 2016. He joined PSU as the Dean of Engineering since Sep. 2016. His research area is in engineering mechanics, materials engineering, tribology, composite and smart materials, metal and ceramic matrix composites, processing of advanced materials, design and modeling of advanced structures, processing of nano-structured materials