American Journal of Engineering Research (AJER)
e-ISSN : 2320-0847 p-ISSN : 2320-0936
Volume-02, Issue-12, pp-194-202
www.ajer.org

Research Paper **Open Access**

Assessment Of Mechanical Properties Of Sintered And Hot Extruded Aluminium And Aluminium Based Titania Composites

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Abstract: **-** The present investigation pertains to evaluate the quality of aluminium and aluminium based titania composite preforms when hot extruded through varying reduction ratios and also asserts their density and mechanical properties. Aluminium powder and powder blend preforms of Al-6% TiO_2 & Al-12% TiO_2 were prepared using 1.0MN Capacity UTM. Preform densities were maintained at 90±1 per cent of theoretical by applying controlled pressure in the range of 290± 10 M Pa and by taking accurately weighed powders. Al-6% TiO_2 and Al-12% Ti O_2 powder blends were separately prepared in a pot-mill. Preforms were sintered under protective coating in an electric muffle furnace for a period of 100 minutes. Extrusion experiments were carried out using 1.0MN capacity UTM while heating the extrusion die and the sintered preform in-situ. Data obtained on extrusion were critically analyzed and properties evaluated were summarized systematically. Addition of Titania in the aluminium matrix enhanced the tensile strength with a little drop in ductility. Enhancement in reduction ratio has been beneficial to mechanical properties.

***Keywords:* -** *composite, extrusion, preforms, properties, sintered*

I. INTRODUCTION

The development of high strength (Powder Metallurgy) P/M materials capable of withstanding elevated temperature service conditions has been the endeavour of the material scientists, physicists and the metallurgists. However, the past nine decades have witnessed the extremely rapid growth of P/M materials (products) that were capable of withstanding severe service conditions were produced mainly through forging or extruding or rolling the sintered P/M billets. This means that when P/M route is suitably combined with the conventional metal forming routes, it provides a more conducive and productive means while blending the powders of distinctly different constituents. These homogeneously blended powders induce into the final products - a uniform distribution of the dispersoid which is a harder phase and can retain its identity which in turn assists the components to retain higher strengths. Such composites cannot be produced by employing any other conventional route of manufacturing. Thus, these products are capable of withstanding high temperatures resulting into easy production of engine components that operate at elevated temperatures. Aluminium being one among the light metals possessing high strength to weight ratio, and, therefore, its applications in automotive industries is on an increasing trend.

The specific advantages of aluminium and its alloys such as light weight, corrosion resistance, high thermal and electrical conductivities, non-magnetic characteristics, variety of forming and finishing operations can be combined with the advantage of powder metallurgy to develop various types of aluminium based composites. Extrusion is reported [1] to be economical when coupled with powder metallurgical route. Basically, extrusion is an act of expulsion of metal by mechanical force [2] through well defined orifice geometries. Mainly, in a hot working operation, the metal is heated to give a suitable degree of softness and plasticity. This process is adopted to develop various types of aluminium based powder metallurgical composites [1].

It is, well known that poly-phase materials are essentially composites, the material distribution here is controlled not mechanically or thermally but, by chemical means. Thus, it is a process of combining materials in certain ways to achieve a desired property which the individual materials would not possess. Aluminium P/M composites are such materials which come in the category of metal based oxides/nitrides/borides/ or their different combinations in metals constitute composites. Such combinations with major constituents being as metal (base) are termed as metal based composites. Their reinforcement particles are of a hard phase which retain their identity by not entering into the matrix (not alloying with the metal/metals) and, thus enhancing the strength, wear resistance etc. These dispersion strengthened materials retain high yield strength or strain hardening rate of elemental or alloy matrices, even at higher temperatures [3]. Composite materials, in general, have been produced by extrusion of thoroughly blended elemental and other hard particles of oxides or carbides or nitrides or borides etc. from powder billets. This is possible when deformation properties of components under extrusion conditions are almost same. This is a universal technique, if compactable combinations of materials are found. Aluminium based composites are ideal for hot extrusion in particular [4]. Hot extrusion is a process used to consolidate metal powders to useful shapes such as solid bars, hollow sections and other unusual geometries. This is a powder metallurgical process offering a large reduction in size from a single operation and the same yields improved densification and enhanced mechanical properties. Thus, the resultant product can be employed for structural applications [5]. The hot extrusion process is widely used for the consolidation of dispersion – strengthened materials in particular SAP and dispersion type of nuclear fuel elements, but the application to pure metal or alloy powders is limited [6]. However, the use of P/M methods to overcome casting problem is proven [7] beyond any doubt. A comprehensive experimental detailed studies and thorough analysis showing the influence of composition, extrusion ratio and the temperature of extrusion is described elsewhere[5]. The dies and tooling used in extrusion are required to withstand considerable abuse from the high stresses, thermal shocks and oxidation problems [8]. It has been shown that the addition of hard particles to aluminium powders have improved both the adhesive and abrasive resistance [9, 10] of the resultant product. Niels Hansen [8] has reported that the properties of dispersion strengthened aluminum products by hot extrusion after powder blending, the strength has gone up and the elongation dropped when matrix aluminium powder particle size was decreased and oxide concentration was raised. The sub – grain structure that was formed in the aluminium matrix during hot extrusion was super imposed on oxide strengthening particles which is effective at elevated temperatures. Some important literature on extrusion of aluminum and aluminium based powder preforms can be referred elsewhere [11-32].

The selection of the systems for the present investigation has been advocated purely on the basis of possible industrial application as the basis of the composite selection. Generally to harden and strengthen the pure copper metal, cold working has been the most adopted process, but, to attain highly stable and strong copper base material can be produced by dispersing non dissolvable second phase material into the matrix of copper and working it hot or cold. Since the composites of aluminum based can not be homogeneously prepared following the conventional melting and casting route, the P/M route was thought to be most appropriate because the virtually insoluble ingredient can be blended, compacted, sintered and extruded. This category is classified as dispersion strengthened aluminum extruded products. The sound metallurgical microstructural features of the composite are likely to enhance the mechanical properties. These materials can be employed as strong structural highly dense products at places where elevated temperature properties are sought upon. Thus, the present investigation is to ensure conclusively the effect of the extrusion temperature, extrusion ratio on the mechanical properties of with and without the addition Titania where two Titania additions; 6% and 12% were made. Properties required to be assessed are tensile strength, per cent elongation and per cent area reduction and also the effect of lubricant employed during extrusion on the quality of the products.

II. EXPERIMENTAL DETAILS

Includes the materials procurements and the types of equipment required for characterization of aluminium powder Al-6% TiO_2 and Al-12% TiO_2 blends, design and fabrication of die set assembly for compaction and also for extrusion dies. Compaction, ceramic coating fabrication of in-situ heating furnace and other relevant details are briefed. Compaction details along with the compaction assembly and also the die plates containing the extrusion orifices are shown. In addition to these, an extrusion assembly is also shown.

II.1 Materials Required

Materials required for the present investigation are commercially pure atomized aluminium powder of -150µm which was procured from M/s The Metal Powder Company Limited, Thirumangalam, Madurai, TamilNadu, India and titanium powder of -38µm was obtained from M/s Ghrishma Speciality Powders, Mumbai, Maharashtra, India. Molybdenum -di- Sulphide paste and graphite powders were also procured from Ghrishma Speciality Powders as stated above. Compaction and extrusion die materials were procured for designing, fabricating and heat treating them to required hardness and toughness. Two extrusion die plates were also fabricated for extruding at an extrusion ratio of 6:1 and 24:1. In- situ heating furnace was also designed and fabricated.

II.2 Equipment Required

Universal Testing Machine of 1.0MN capacity was required for powder compaction and powder preform extrusion. Separate electric muffle furnace was required for sintering the compacts. An electronic balance with a sensitivity of 0.0001g was required for density measurements. Apart from this temperature controller cum indicator along with chromel / alumel thermocouple was also required. Lathe machine for tensile specimen preparation was needed along with the Haunsfield Tensometer for conducting tensile tests and other measuring devices such as electronic vernier calipers, etc.

II.3 Preparation of Titania Powder

A known amount of Titanium powder of -38 µm was spread in a stainless steel tray and the tray was kept in an electric muffle furnace maintained at 1273±10K and allowed the powder to oxidize for a period of two hours and cooled to room temperature. This oxidized powder was ground in a porcelain bowl with a porcelain stirrer manually. This operation was continued till the titanium powder was completely oxidized, and, ground to quite fine sizes, i.e., less than 38µm. This prepared Titania powder was used to prepare Al-6%TiO_2, and, Al-12%TiO_2 composite powder blends. Chemical analysis revealed that in the prepared TiO_2 powder, the titanium content was found to be exactly in stoichiometric composition of TiO_2.

Table: 1. Characteristics of Al Powder, Al-6%TiO_2 and Al-12%TiO_2 Powder Homogeneous Blends

System	Properties Evaluated		
	Apparent Density, g/cc	Flow Rate S/100g	Compressibility, g/cc at a Pressure of 290±10MPa
Al	0.9308	60.37	2.430
Al-6%TiO_2	0.9398	58.3	2.491
Al-12%TiO_2	0.9698	56.7	2.549

Table: 2. Sieve Size of Aluminium Powder

Wt. %	Sieve Size									
	-180 +150	-150 +126	-126 +106	-106 +90	-90 +75	-75 +63	-63 +53	-53 +45	-45 +38	-38
Wt% Ret.	1.60	3.60	2.50	0.71	8.30	9.20	16.70	15.80	3.63	37.95
Cum Wt% Ret.	1.60	5.20	7.70	8.41	16.71	25.91	42.61	58.41	62.04	99.99

II.4 Powder Blend Preparation

Known amount of Al-6%TiO_2 and Al-12%TiO_2 powder mixes separately were taken into two different stainless steel pots with powder mix to porcelain balls (~ 10 to ~20 mm diameters) weight ratio and the lids of the pots were securely tightened and placed on the pot mill. The blending operation was carried out for a period of 30 hours so as to obtain homogeneous powder blends. Homogeneity was confirmed by taking 100g of powder mix after every one hour blending for measuring apparent densities and flow rates. Immediately after the completion of each test, the powder mixes taken out were returned back to their respective pots and lids were tightened again. This process was repeated till the last three consecutive readings of flow rates and apparent densities were found to be consistently constant and, thus, the time of blending was found out to be 30 hours.

II.5 Compact Preparation and Application of Ceramic Coating

Green compacts of aluminium, Al-6% TiO_2 and Al-12% TiO_2 powder blends were prepared by using suitable die, punch and bottom insert on a 1.0MN capacity Universal Testing Machine. The compacts of 27.50 mm diameter and 32.00mm height were prepared by taking pre- weighed powder and or powder blends and pressed into a density range of 89 ± 1 per cent of theoretical by applying controlled pressure in the range of 345± 10 M Pa. The powder compaction die set assembly is shown in Fig. 1. Indigenously developed and modified ceramic coating[33] was applied on the entire surfaces of the green compacts of all compactions while maintaining their identity and allowed them to dry under an ambient conditions for a period of 12 hours . A second coat was applied 90^o to the previous coating and allowed them to dry under the aforementioned conditions for a further period of 12 hours.

These ceramic coated compacts were placed in a stainless steel tray and the complete set was transferred into the furnace chamber maintained at 473±10K. Compacts were dried at this temperature, for a period of half- an- hour and then were allowed them to remain in the furnace itself for sintering operation.

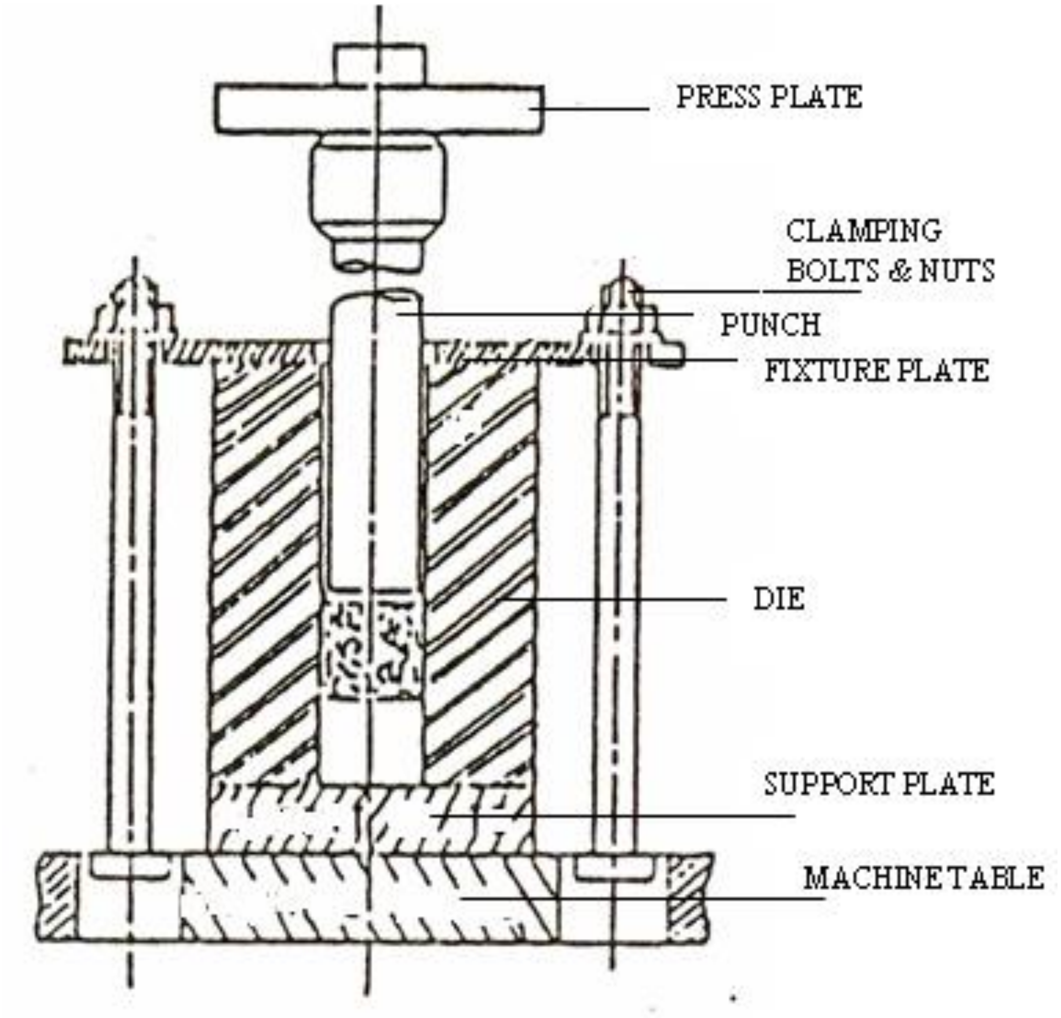

Figure 1 Compaction Assembly Showing complete Details

II.6 Sintering

Ceramic coated compacts were sintered in an electric muffle furnace for a period of 100 minutes in the temperature range of 823±10K. All sintered compacts were cooled inside the furnace itself by switching off the furnace. Instead of furnace atmosphere being hydrogen, dissociated ammonia or nitrogen, the compacts were coated with the indigenously developed and modified ceramic coating which protected the compacts against oxidation during sintering. The ceramic coating was tested upto 1473±10K and was found to be non-permeable to air or other gases while sintering ferrous based preforms at the aforesaid temperature. Therefore it was presumed that the ceramic coating applied over the compacts was highly protective during sintering at 823±10K as well.

II.7 Hot Extrusion

All hot extrusion experiments were carried out by using the suitable die- set assembly along with the in-situ heating furnace. Figure-2(a) shows the die plates with two different openings for hot extrusion. The entire die-set components were designed, fabricated using hot die steel, suitably heat treated to 55-58Rc values and finally tempered to retain the hardness in the range of 48-52 RC values. All hot extrusion experiments were carried out at two different extrusion temperatures; 773 ±10K and 823 ±10K and at two extrusion ratios namely,

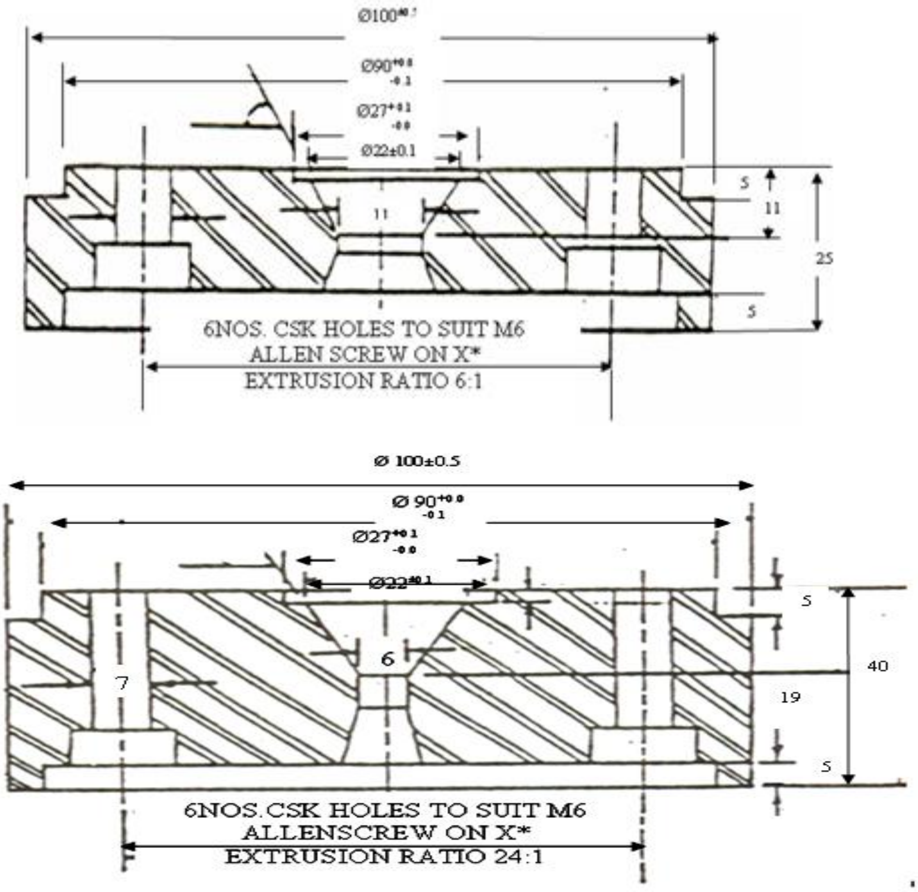

Figure 2(a) Die Plates for Extrusion with 6:1 and 24:1 Extrusion Ratio with Dimensions in mm.

6:1and 24:1 respectively. The press used for extrusion was 1.0MN capacity Universal Testing Machine. Die plates are shown in Fig. 2(a) and Fig. 2(b) shows the entire extrusion assembly along with the heating arrangement (in-situ heating provision).

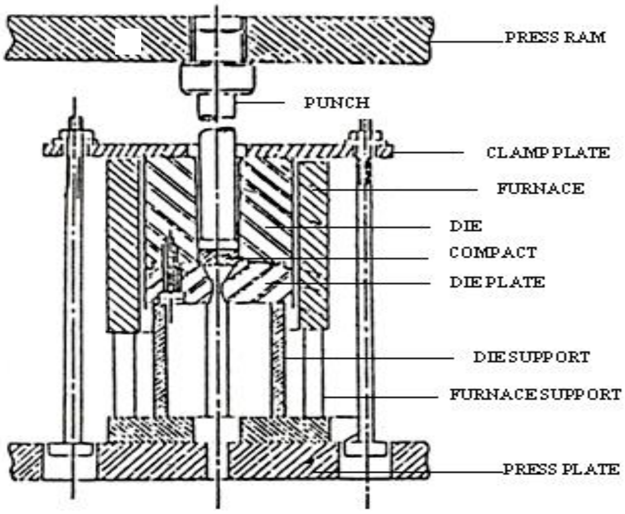

Figure 2(b) Complete Extrusion Assembly Along with the in-situ Heating Arrangement

II.8 Tensile Testing

Required length of tensile pieces were cut from each of the extruded rods from both extrusion ratios and both extrusion temperatures and the same were machined to standard tensile specimens as described elsewhere [34]. Prior to carrying out the tensile testing, the density measurements were carried out by using methods detailed elsewhere [35]. The masses in air and water were measured on a single pan electronic balance of sensitivity, 0.0001g [36]. Tension tested pieces were carefully used to calculate the final dimensions such as necked diameter and the final gauge length of the broken specimen.

III. RESULTS AND DISCUSSION

Results of all extrusion experiments were consolidated and were discussed in detail. This further includes recording of the pressures to begin and to end the extrusion for each extrusion at both temperatures of extrusion and at both extrusion ratios. All extrudes were visually observed and found that their general surface appearances were in two categories; (a) Good, and, (b) Excellent. Further, the densities attained at every extrusion temperature and extrusion ratios with their tensile properties are also recorded. During all extrusion experiments a paste consisting of molybdenum – di – sulphide and graphite was employed as a lubricant in order to reduce the frictional constraints while carrying out extrusions.

III.1 Effect of Lubricant on Extrusion Quality

Extrudes obtained at each of the extrusion ratios and the temperatures of extrusions were visually observed, and, found that, in general, the surface appearance were good to excellent. This observation is summarized in Table –3. This table reveals that in case of aluminium extrudes the surface quality was categorized as good whereas in case of extrudes of Al -6% TiO_2 and Al-12%TiO_2, the surface quality at all temperatures of extrusions and extrusion ratios were categorized as an excellent surface finish.

Table: 3. Surface Finish of the Extrudes of Aluminium, Al-6% TiO_2 and Al-12%TiO_2 at Both Extrusion Temperatures and Ratios

System composition	Lubricant used	Temperature of extrusion , K	Extrusion Ratio	Quality of Surface Finish
Al	MoS_2 + Graphite Paste in Acetone	773	6:1	Good
			24:1	Good
		823	6:1	Good
			24:1	Good
Al-6% TiO_2	MoS_2 + Graphite Paste in Acetone	773	6:1	Excellent
			24:1	Excellent
		823	6:1	Excellent
			24:1	Excellent
Al-12% TiO_2	MoS_2 + Graphite Paste in Acetone	773	6:1	Excellent
			24:1	Excellent
		823	6:1	Excellent
			24:1	Excellent

III.2 Effect of Experimental Parameters on the Final Density Attained With Beginning and Ending Extrusion Pressures

Table-4 shows the effect of extrusion ratios and the extrusion temperatures on the pressure required to begin the extrusion and also the pressure at final stages of extrusion. It is observed from this Table –4 that at constant extrusion temperature and constant extrusion ratio, the pressure to begin extrusion has gone up as the Titania content from 0.0% to 12% was raised. For instance at extrusion ratio of 6:1 and at the extrusion temperatures of 773K, the pressures to begin extrusion is in an increasing order such as 126 M Pa, 152M Pa and 160M Pa for aluminium, Al-6% TiO_2 and Al-12%TiO_2 respectively. Similar is the case for the ending pressure. The above is true at both higher extrusion temperature and higher extrusion ratio. Once the extrusion ratio is raised, the value of pressure to begin extrusion has gone up. This is true for all compositions at both extrusion temperatures. Further Observing the Table-4, the percentage density achieved is highest when the extrusion temperature and the extrusion ratio both were greater. It is, further observed that at extrusion ratio of 24:1 and

Table: 4. Effect of Experimental Parameters such as Temperature and Extrusion Ratios on the Final Achieved Density

System Composition	Temperature of Extrusion, in K	Extrusion Ratio	Pressure, in MPa		%Density Achieved
			Beginning	Ending	
Al	773	6:1	126	139	98.58
		24:1	209	286	99.60
	823	6:1	105	128	99.22
		24:1	130	215	99.71
Al-6%TiO_2	773	6:1	152	165	98.59
		24:1	250	300	99.73
	823	6:1	125	142	98.76
		24:1	230	285	99.73
Al-12%TiO_2	773	6:1	160	190	98.97
		24:1	298	315	99.30
	823	6:1	153	170	98.98
		24:1	235	250	99.57

the extrusion temperature was 823K, the density attained in each extrudes has been beyond 99 per cent of theoretical, hence, the properties are expected to be quite enhanced and in fact they were fairly high.

III.3 Effect of Composition, Temperature and Extrusion Ratios on Mechanical Properties

Table-5 shows the percentage density achieved and tensile properties such as ultimate tensile strength, per centage area reduction and per centage elongation showing the influence of extrusion ratio, extrusion temperature and system composition. It is observed that irrespective of the extrusion temperature and the extrusion ratio, as the titania content in aluminium is raised from 0.0 to 12 per cent, the values of ultimate tensile strength have gone up down the column, but, reverse is true for per cent elongation and per cent area reduction. It is further observed that

Table: 5. Effect of Extrusion Temperature, Extrusion Ratio and Composition on Attained Per cent Density and Mechanical Properties

Temperature of Extrusion, in K	System Composition	Extrusion Ratio							
		6:1				24:1			
		%(ρ_f/ρ_{th})	UTS, MPa	%El	% A.R.	%(ρ_f/ρ_{th})	UTS, MPa	%El	% A.R.
773	Al	98.58	128	15.0	13.40	99.60	154	18.4	16.53
	Al-6%TiO_2	98.59	200	14.10	10.60	99.73	210	16.01	13.06
	Al-12%TiO_2	98.96	216	9.60	9.20	99.30	229	11.10	9.98
823	Al	99.22	144	16.4	13.8	99.71	176	18.60	17.41
	Al-6%TiO_2	98.76	206	14.81	11.01	99.73	220	17.41	14.38
	Al-12%TiO_2	98.98	232	10.13	10.13	99.57	243	13.40	11.43

As the extrusion ratios and the extrusion temperatures were raised, the attained densities and the attained tensile strengths have gone up. This clearly establishes that at higher extrusion ratios and higher extrusion temperatures, the coherency of mass and bond formation, both have enhanced. The addition of Titania in aluminium as dispersoid has though dropped toughness as is indicated by per cent area reduction and per cent elongation, but, this drop is only marginal compared to the rise in ultimate tensile strength values. Thus, the present investigation leads the way for the future production of aluminium based composites for structural applications.

IV. CONCLUSIONS

Based on the experimental data obtained and the calculated parameters including their critical analysis led to the following major conclusions to be drawn from the present investigation:

1. Extrudes obtained possessed very smooth surface finish due to the use of molybdenum – di- sulphide and graphite mixed lubricant during extrusion. The surface finish has been categorized for aluminium as good and Excellent for Al – 6%TiO_2 and Al -12%TiO_2 systems,

2. Addition of titania in aluminium matrix established beneficial effect in enhancing the tensile strength even though a mild drop in per cent area reduction and per cent elongation ,i.e. a little drop in ductility which is a measure of toughness and hence a little drop in toughness,
3. An increase in extrusion ratios and the extrusion temperatures and their combination along with the lubricant have produced strong coherent mass with increase in per centage elongation and per centage area reduction meaning thereby an increase in ductility and so as the toughness.
4. Pressure required to begin the extrusion has been established to increase when the extrusion ratio was higher and extrusion temperature was lower. Same effect has been established by an increase in Titania contents in aluminium matrix.
5. Addition of titania in aluminum matrix has established an increase in both the pressure required to begin extrusion and the pressure at the end of extrusion, and,
6. High level of directionality of Titania dispersoid along the direction of extrusion is an anticipated possibility when the extrusion ratio and extrusion temperatures are raised.

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