Investigation on Cold Extrusion of Aluminium - Aluminium Oxide Powder Composite Materials

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ABSTRACT

Cold-sintering process was studied by investigating both the extrusion variables and composite variables. The extrusion variables are; the extrusion velocity, the extrusion ratio, the extrusion die angle and the relative billet size. The composite variables are; the oxide fraction and the oxide size. Three processes of powder consolidation including hot extrusion process (B), hot-sintering process (D) and vacuum-hot-pressing process (F) were compared with the cold-sintering processes including de-gassing of powders (process C) and without de-gassing (process A). A modified process was developed to bond aluminium and aluminium oxide phases by thermo-compression-welding process (E) and compared with the other consolidation processes.

The results of this work showed that the cold-sintered products require small extrusion pressure and possess high tensile strength and hardness. De-gassing of billets before extrusion gives composites with high density and allows the composites to be heat treated at temperatures lower than the de-gassing temperature without any loss of the composite density. The impact toughness is very sensitive to the small changes in the composite density. The composite strength, hardness and ductility can be expressed and estimated by empirical equations from the structure parameters. Decreasing the oxide fraction of composites with non-coherent oxides (process A) improves the wear resistance and therefore the toughness and ductility determines the wear behaviour. Increasing the oxide fraction in coherent two phases composites (process E) improves their wear resistance, the hardness of the composite controls the wear behaviour.

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CONTENTS

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NOMENCLATURE

A	Cold extrusion, consolidation process.
A_v	Impact energy (Joule).
A_5	Elongation (%).
B	Hot extrusion, consolidation process.
C	De-gassing+cold extrusion, consolidation process.
D	Hot sintering+cold extrusion, consolidation process.
D_b	Billet diameter (mm).
d	Oxide particle size (μm).
E	De-gassing+hot densification+cold extrusion (thermo-compression welding), consolidation process.
f	Oxide volume fraction (%).
F_p	Punch force (MN).
F_d	Die force (MN).
F_f	Friction force (MN).
F	De-gassing+vacuum hot compaction+cold extrusion, consolidation process.
HV 10	Vicker's hardness.
H	Material hardness.
M	Billet weight (g).
m	Oxide weight fraction (%).
P	Extrusion pressure (N/mm^2).
p	Contact pressure in wear test (N/mm^2).
R	Extrusion ratio.
R_m	Ultimate tensile strength (N/mm^2).
$R_{p0.2}$	Yield stress (N/mm^2).
s	Wear sliding distance (km).
T	Temperature (K).
t	Time (hour).
V	Sliding velocity in wear (m/s).
v	Extrusion velocity (mm/s).
w	Weight-loss in wear test (g).
$\dot{w}$	Wear rate (mg/km or cm^3/cm).
$\dot{w}^{-1}$	Wear resistance = 1 / wear rate, (cm/cm^3).
Z	Reduction in area (%).
α	Extrusion die angle (^{o}).
γ	Relative density (%).
λ	Mean-free-path (μm).

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CHAPTER 1

INTRODUCTION

Cold-sintering is the consolidation of powder particles by plastic flow in gradients of high pressure leads to high density, physical contact and chemical bonding of freshly surfaces at room temperature. One of the main advantages of cold-sintering may be the design and production of composite materials, especially in cases of systems which are unobtainable (or difficult to obtain) by conventional metallurgical means or by conventional PM methods, because of decomposition or dissolution of one of the phases, or because of stringent processing requirements. Cold-sintering may be used for production of metal-carbide, metal-nitride and metal-oxide composites, as well as metal-metal composites, especially in the cases when there is no mutual solubility between the components.

The cold-sintering technique of powders has opened a way to produce materials with significant better properties of tensile strength, fatigue strength and strength at elevated temperatures compared to the properties of materials made melting metallurgically or powder metallurgically. It was possible to manufacture compact Al-materials by cold-sintering (cold extrusion) of Al-powders without sintering treatment at laboratory and industrial scales. The results showed the same tendency with a distinction in the values. The strength properties were superior to the properties of Al99.5, and the toughness properties were satisfactory.

Powder composites were produced on the laboratory scale by cold extrusion of aluminium-aluminium oxide powder mixtures, that contain up to 30 wt.% oxide. Static and dynamic strength data decreased with increasing fraction of oxide powders, but the absolute values exceed the corresponding values for Al99. The wear resistance is a function of amount and size of the

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oxide particles. The wear resistance of the composites improved as the hardness of the counterfaces is increased.

The aim of this work is to study the possibility of producing aluminium-aluminium oxide composites by cold-sintering on the industrial scale without deteriorating the strength properties and wear resistance. The possibility of improving the composite properties by de-gassing the powders or by welding the two phases has to be considered. The strength properties as well as the wear behaviour of specimens consolidated by cold-sintering worth to be compared with other consolidation processes.

To achieve the above mentioned objectives cold-sintering process is to be investigated in scope of both the extrusion variables and composite variables. The extrusion variables are; the extrusion velocity, the extrusion ratio, the extrusion die angle and the relative billet size. The composite variables are; the oxide fraction and the oxide particle size. The different consolidation processes are to be compared. A detailed study of the strength properties and wear behaviour of $Al\text{-}Al_2O_3$ composites are to be investigated.

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CHAPTER 2

LITERATURE REVIEW

Plastic flow of powder particle in gradients of high pressure leads to high density, physical contact and chemical bonding of freshly formed surfaces at room temperature. GROSCH and JÄNICHE[1] found that it was possible to manufacture compact aluminium materials by cold extrusion of Al-powders without sintering treatment if the extrusion ratios greater than 8.35:1 are applied. The mechanical properties of this material (tensile strength R_m and rotary bending strength σ_{bw}) were superior to that of Al99.5 and the toughness was satisfactory. The powders with coarse particle size could be extruded with lower extrusion pressures and possess higher ductility with lower strength as compared to the fine particle extrudate. Investigations on both the experimental and industrial extrusion presses showed a distinction in the values, however, the results showed the same tendency.

GROSCH and BROCKMANN[2] succeeded in cold extruding Al- and Al_2O_3- powder mixtures up to 35 wt.% Al_2O_3 to homogeneous composite materials without prior sintering treatment using experimental extrusion press. The static strength properties of the composites were function of the oxide fraction. The strength and toughness decreased with increasing the oxide fraction. Although the fatigue strength of the composites was influenced by the dispersion of the oxide, it did not sink below the level of the treated Al99.5. On the other hand, the wear resistance of Al-Al_2O_3 composite had proved to be excellent.

2.1 Cold-Sintering of Powders:

The process of consolidating powders under pressure to full density without applying heat is called "Cold-Sintering". Earlier, the powder was taken in the as-recieved condition and mixed with ~1% of solid lubricant such as zinc stearate, primarily

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to decrease friction against the die walls. It was formed into a green compact by pressing at a load greater than the yield stress of a comparable specimen of compact material. Subsequent sintering was required to bind the area already in contact, but not welded, and to evaporate the solid lubricant.

Now, cold sintering takes place by cold compaction to green billets followed by one of the densification methods as: high pressure cold pressing, cold rolling, high energy rate forming, cold isostatic pressing and cold extrusion.

Cold sintering of powders under pressure is widespread to many metal powders such as aluminium[1,2] and stainless steel[3,4], refractory metal powders such as V, Nb, and Ta[5], and to mixture of metal/ceramics[6,7]. Generally, good quality of cold sintering (high density, high mechanical properties and high geometrical and dimensional accuracy) was obtained for materials that can easily be deformed plastically under pressure. The energy consumption in cold sintering process is relatively lower than the other consolidation processes including hot sintering.

There are many factors that can affect the extrusion pressure, the mechanical and physical properties of the cold sintered parts:

The Extrusion Velocity was found to have an effect on the extrusion pressure. PUCH[10] found a decrease in the extrusion pressure of 15% when the velocity increased from 0.17 to 3.40 mm/s, while WILLIS and BRYANT[11] found no perceptible effect in the higher range of velocities (270 to 500 mm/s). A minimum pressure was observed by GROSCH and JÄHICHE[2] in the velocity range of 10 to 20 mm/s. The decrease in pressure was attributed to the heat generated during extrusion. SHEPPARD[12] estimated that 90% of the work-done is converted to heat energy. It was found that the higher the velocity, the smaller will be the tensile strength and yield stress of the extrudate[2,10].

The Extrusion Ratio was found to have an effect on the powder densification. It was possible to have compacted Al-material

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when the extrusion ratios are greater than 8.35:1[1] and 10:1[13] . The pressure increases logarithmically with increasing the extrusion ratio[2,10,14].

$$P = a + b \ln R \quad \text{................(2.1)}$$

where: R is the extrusion ratio,
a,b are constants.

GROSCH and JÄNICHE[1] found that increasing the extrusion ratio has led to an increase in the tensile strength using a laboratory press with small extrusion velocity. The tensile strength decreased using an industrial press at higher velocities. It could be attributed to the heat generated during extrusion at higher velocities. RUPPIN and MÜLLER[14] emphasized the second result.

The Extrusion Die Angle whose optimum gives a minimum extrusion pressure are given in literature[8,11,15,16,17]. The extrusion ratio and the friction conditions affect the value of this angle. The greater the extrusion ratio or the greater the coefficient of friction,the greater the optimum angle. Avitzur[16] put the following relation to calculate the optimum die angle based on the Upper-Bound-Theorum:

$$\alpha_{opt.} = \sqrt{3 f \ln R} \qquad (2.2)$$

where: f is the friction factor, which can be estimated from the Ring-Compression-Test[16]

JÄNICHE[2] found that the larger the die angle, the smaller will be the tensile strength, yield stress and ductility too.

The Oxide Fraction that when increased in the composites led to decrease the strength properties of the composites[6,18]. This was attributed to the fact that the hard oxide do not contribute to strength and higher oxide fractions reduce the stressed cross-section at constant specimen cross-section. The ductility of the composites decreased with increasing the oxide fraction.

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The Oxide Particle Size: The decrease in particle size of the second-phase led to an increase in the required extrusion pressure[9] and the strength properties of $Al-Al_2O_3$ composite material[18,19]. The particle size of the oxide has no effect on the composite ductility[18].

2.11 Bonding Mechanism in Cold Sintering:

2.111 The Welding of Aluminium Particles:

JÄNICHE[2] considered the cold sintering as cold pressure welding of the metal powder particles. Hitherto, there is no general acceptable theory to describe the bonding mechanisms in cold pressure welding. It was thought that *Diffusion* was the controlling process[20] due to the heat generated during deformation. The diffusion hypothesis did not appear to be adequate because it was possible to weld aluminium at liquid air temperature[21]. It was proposed that welding was controlled by *The Break Up of Surface Oxide*[22]. Initially, this hypothesis seemed to provide a correlation with weldability, i.e. the different weldability of different metals was attributed to the relative hardness of the bulk metal and oxide[23,24]. This is due to the initiation of welding as controlled by the degree of fragmentation of the oxide film. Subsequent work showed this hypothesis to be unsatisfactory[25]. An alternative hypothesis, the essential process in cold welding is that *Overcoming Surface roughness* of the contacting surfaces in order to obtain interfacial contact[26,27].

The present tendency is to think in terms of an "*Energy Barrier*" which controls welding, and that increased temperature, or prior cold working, help to overcome this barrier[28,32]. The energy barrier arises either from the necessity to reorient surface atoms to form a bond, or from the work required to disperse contaminant. It was thought that the energy barrier was recrystallization[33] and diffusion[31]. SEMENOV[28] suggested that this energy comes from misorientation of the crystal at

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