MODELLING OF SINTERING BEHAVIOUR OF Al-49.6wt%Zn POWDER COMPACTS

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ABSTRACT

Al-Zn alloys find commercial use in aircrafts and cars. During multiphase sintering of powders, dimensional changes take place. It is necessary to be able to accurately predict what dimensional changes will occur so that post sintering operations will take care of this change. The sintering behaviour of Al-49.6wt%Zn pbwder compacts has been studied. Mathematical models for the effect of pressure and temperature on dimensional changes during sintering were obtained. Sintering temperature was kept constant at 435^{0}C. Powder compact size was also kept constant. It was observed that, at a compacting pressure of 288Mpa there was zero shrinkage after 6 minutes of sintering at 435^{0}C.With compacting pressure less than 288Mpa and sintering for 150minutes there was no single pressure where both diametric and longitudinal shrinkage was zero. It was only possible to keep the longitudinal and diametric shrinkage as low as possible (0.35%) at a compacting pressure of 205MPa . This shows that it is possible to have no shrinkage in the sintering operation of Al-49.6wt.%Zn powder compacts which implies that no post sintering operation is necessary if dimensional accuracies are required.

Key words: Modelling, Sintering, Al-49.6wt.%Zn, Powder-compacts.

INTRODUCTION

Dimensional changes occur during sintering of metal powder compacts such that the compact may measure undersize or oversize(Brophy et al,1966; De Garmo,1979 and Yankee,1979). This is a result of myriad of factors including sintering time, compacting pressure, sintering temperature, particle size shape and powder mix. Generally, the part tends to increase in density as sintering proceeds, which leads to shrinkage. However, growth may occur due to diffusion effect[3] - a result of powder mix of different elements(Vlack,1989). This growth is most marked during multi-phase sintering as in this research work. The presence of grain boundaries near pores also permits densification of material to continue (Brophy et al,1966).

Sintering leads to progressively increased strength by causing particles to weld together by diffusion[4]. Mechanical properties are improved as densification progresses. Keeping dimensional changes to the barest minimum help in subsequent post sintering operation thereby keeping cost to the barest minimum.

Modelling of sintering behaviour is of great interest[5] to researchers today. Research efforts are directed in three fronts:

Shrinkage behaviour of sinters which consist of Powders which exhibit almost isotropic shrinkage on sintering and Powders exhibiting anisotropic shrinkage on sintering ; micro mechanical modelling to improve knowledge of the constitutive equations ; and modelling of particle rearrangement during sintering.This work falls in the first group - to investigate the shrinkage during sintering in Al-49.6wt%Zn powder compacts.

EXPERIMENTAL PROCEDURE

Materials and Materials Preparation

The aluminium powder used in this project was obtained from NIPOL Nig. Ltd, Ibadan, and manufactured by Norddeutsche Addinerie, Hamburg, Germany. It is commercially pure Al of 98% purity. The zinc powder was obtained from the Metallurgical and Materials Engineering Department laboratory, Obafemi Awolowo University, Ile-Ife. Sizes of the powders were determined by mechanical sieving machine. The Al powder particles were in the range of 4.5 um in linear dimensions, while the Zn particles were slightly larger, but less than 12.5 um in linear dimensions.

The weighing balance used for weighing the powder components was the Metler chemical balance with a maximum mass capacity of 151 g. Stearin was used for die wall lubrication. Rigid steel tool (die and plunger) was used for compaction. The cylindrical die had inner and outer diameters of 2.55 and 4.25 cm, and was 9.8 cm long. The powder pressing equipment used was the hydraulic laboratory press with a maximum pressing capacity of 19.5 metric tonnes. The sintering operation was carried out in the laboratory electric tubular furnace capable of heating up to a temperature of 1500^0C. Measurement of compact dimensions was carried out with vernier callipers with an accuracy of

0.095cm. Etchant was a mixture of 25 ml HNO_3 and 75 ml H_2O for phase identification.

Methods

Effects of sintering time and compacting pressure on dimensional and volumetric shrinkage of the powder compacts were carried out.

Variation of volume shrinkage with sintering time

50.4g of Al powder was added to 49.6g of Zn powder and hand mixed. The mixed powder was loaded into the die and compacted. Prior to compaction, the die walls and plunger surface were lubricated with stearin. The powders were compacted under the press at 288 MPa. The compacts were left under the press for about 30 seconds before release and then ejected by pressing the plunger downwards until the compacts came out through the other end. The initial dimensions (thickness and diameter) were measured using vernier calipers. The compacts were sintered for varying times from 0.5 to 150 minutes at 435^{0}C(Dowson,1999). The final thickness and diameter were then measured and recorded. The result is presented in Table1.

Variation of volume shrinkage with compacting pressure

100 g mix of aluminium and zinc in a weight ratio of 50.4:49.6 was made. The die walls and plunger surface were lubricated with stearin. The mix was loaded into the die and compacted in batches. Twelve compacts were made under differing loads from 96 - 365 MPa (the die had a diameter of 2.55 mm) and ejected. They were then sintered for 150 minutes in the tubular furnace. The initial dimensions (prior to sintering) and final dimensions were taken and recorded. The result is summarized in Table2.

Metallography

Four of the compacts were picked for metallographic examination. They were the specimens compacted at 96, 192, 288 and 365 MPa. The specimens were ground using the hand grinder in the MME laboratory with emery paper of 200, 400, 550 and 600 fine. The ground specimens were polished with an alumina emulsion with a hand polisher. After polishing, the specimens were etched by immersing in the etchant (25 ml HNO_3 and 75 ml H_2O) for 40 s at 70^{0}C (ASTM,1972) and rinsed in cold water. The specimens were then examined under a light microscope at a magnification of X100. The results obtained are shown in Plates A-D.

Mathematical modelling

Values for initial and final diameter and thickness powder compacts were imputed into a C++ program. The program calculates the linear and volume shrinkage for each compact along with the log value of time/compacting pressure (negative values for shrinkage indicates a growth). The program then prints these values into a Microsoft™ Excel workbook. Using the chart wizard in Microsoft Excel™, the line of best fit is plotted as well as the R^2 value for the plot. For experiment investigating shrinkage and log of time, from the R^2 value, it was discovered that a polynomial trend was most appropriate. For the experiment investigating shrinkage and compacting pressure, it was discovered that a linear trend/regression type was most appropriate. The line of best fit created is the least squares fit of the XY plot. R^2 values were generated using a transformed regression model of the data values.

$$R^2 = (Y_j - Y'_j)^2 / (Y_j^2) - (Y'^2_j)/n$$

Where Y is the value on the Y-axis

Y' is the average value of Y

n is the number of samples

RESULTS AND DISCUSSIONS

Results

Table 1 shows the shrinkage values obtained during sintering with varying sintering temperatures:

Discussion

Figures 1-3 show the shrinkage pattern with increasing sintering temperature.

The relationship between longitudinal shrinkage and sintering time as a linear one. The equations are presented below.

1. For volume shrinkage (S_v):

   $S_v = -8.4983t+18.723$

   $R^2 = 0.6988$

2. For shrinkage in thickness (S_t):

   $S_t = -7.0324t+15.629$

   $R^2 = 0.6151$

3. For shrinkage in diameter (S_d):

   $S_d = -0.6726t+1.426$

   $R^2 = 0.7741$

Figures 4- 6 show the relationship between, shrinkage and compacting pressure as a linear one and is given by:

1. For volume shrinkage (S_v):

   $S_v = 0.0769\,P + 16.726$

   $R^2 = 0.6186$

2. For shrinkage in thickness (S_t):

   $S_t = 0.0538\,P + 11.131$

   $R^2 = 0.5386$

3. For shrinkage in diameter (S_d):

   $S_d = -0.0115(P) + 2.8281$

   $R^2 = 0.5509$

   P is pressure in MPa

   t is time in seconds

R^2 values were generated using a transformed regression model of the data values. R^2 values indicate a better correlation when less than 1.

From the photomicrographs (plates, A - D) the aluminium-rich (light) and Zinc-rich phases can be seen. The micrographs reveal interparticle diffusion at high compacting pressures whereas there is more of pore filling at lower compacting pressure of 96 Mpa

CONCLUSION

The sintering behaviour of Al-29at%Zn powder compacts has been studied. Mathematical models for the effect of pressure and temperature on dimensional changes during sintering were obtained. Sintering temperature was kept constant at 435^{0}C. Powder compact size was also kept constant. It was observed that, at a compacting pressure of 288Mpa there was zero shrinkage after 6 minutes of sintering at 435^{0}C.with compacting pressure less than 288Mpa and sintering for 150minutes there was no single pressure where both diametric and longitudinal shrinkage was zero, it was only possible to keep the longitudinal and diametric shrinkage as low as possible (0.35%) at a compacting pressure of 205Mpa.

From this findings, Al-49.6wt%Zn powder compacts can be manufactured by powder metallurgy method to close dimensional accuracies without post sintering operations.

REFERENCES

ASTM (1972) "ASTM Book of Standards Part 31, Metals " 1972, ASTM, Philadelphia.

Brophy J. H.; Rose M. R. and Wulff J. (1966)" The structure and Properties of Materials" Vol. 2 John Wiley and Sons.

De Garmo P. (1979) `Materials and Processing in M Manufacturing; 5th Edition , New York, Macmillan Publishing Co.

Dowson G. (1999) "Introduction to Powder Metallurgy". The Process and its Product; 1999 European Powder Metallurgy Association

Yankee.Y. 979) "Manufacturing Processing"; New Jersey Prentice Hall Inc.

Table 1: Dimensional changes of sintered Powder Compact with varying sintering time

Initial diameter (cm)	Final diameter (cm)	Initial thickness (cm)	Final thickness (cm)	Time (mins)
2.53	2.54	0.65	0.67	0.5
2.515	2.51	0.5	0.54	1.2
2.5	2.5	0.7	0.7	4
2.5	2.5	0.585	0.58	6
2.55	2.54	0.655	0.635	10
2.54	2.535	0.66	0.6	18
2.58	2.56	0.76	0.645	35
2.57	2.535	0.55	0.5	48
2.57	2.55	0.68	0.645	90
2.57	2.535	0.545	0.5	150
2.565	2.535	0.54	0.5	60

Table 2: Dimensional shrinkage in Al-29at%Zn Powder Compacts with variation in Compacting Pressure

Initial diameter (cm)	Final diameter (cm)	Initial thickness (cm)	Final thickness (cm)	Compacting pressure (Mpa)
2.585	2.55	0.92	0.92	5
2.525	2.485	0.695	0.63	6.5
2.55	2.525	0.61	0.6	7.5
2.55	2.54	0.595	0.59	8.5
2.5	2.5	0.64	0.62	10
2.545	2.475	0.55	0.55	11.5
2.53	2.53	0.62	0.625	12.5
2.54	2.54	0.61	0.62	13.5
2.57	2.535	0.545	0.5	15
2.53	2.53	0.46	0.48	16.5
2.525	2.57	0.4	0.465	17.5
2.52	2.56	0.54	0.545	19

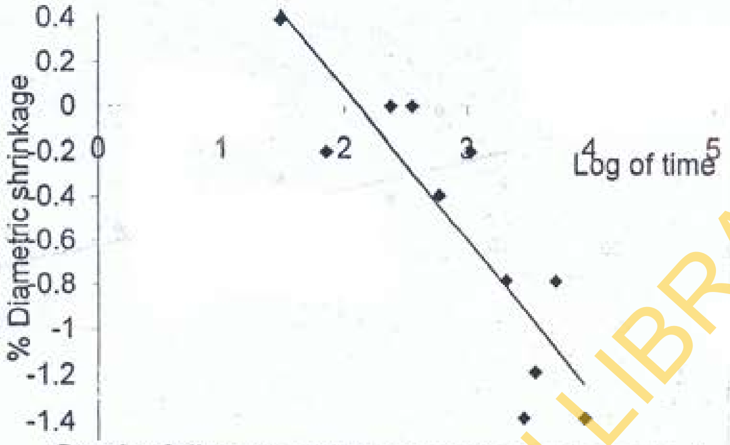

Figure 1: Graph of diameter shrinkage with varying sintering time

Figure 2: Graph of % change in longitudinal shrinkage with varying sintering time

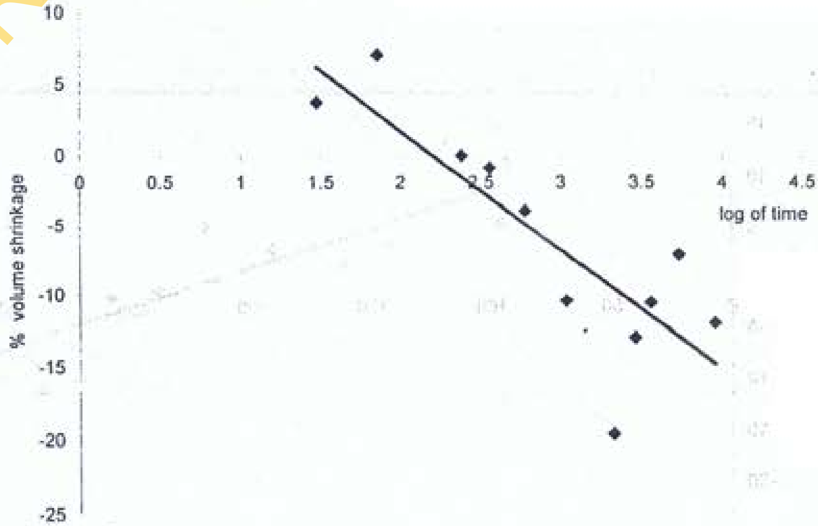

Figure 3: Graph of % change in volume with sintering time

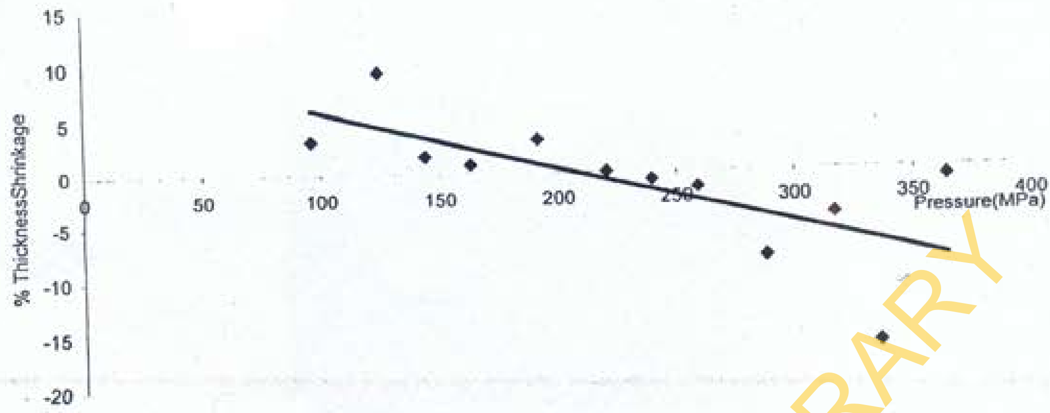

Figure 4: Graph of % thickness shrinkage with varying compaction pressure

Figure 5: Graph of % diameter shrinkage with varying compaction pressure

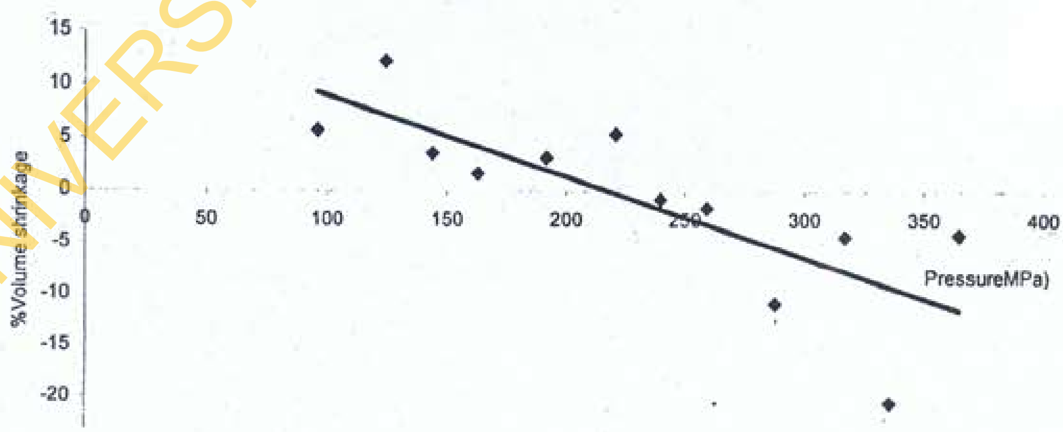

Figure 6: Graph of % volume shrinkage with varying compaction pressure

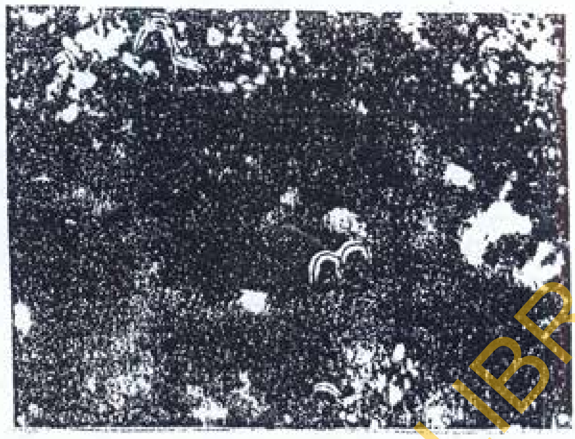

Plate C: **Sample compacted at 288 Mpa (X 100, etched in 25% HNO_3)**

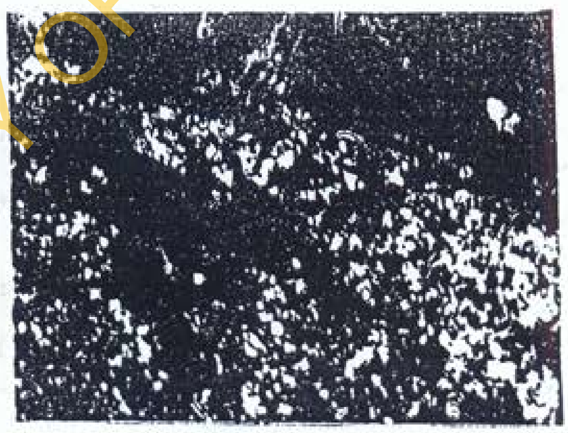

Plate D: **Sample compacted at 365 Mpa (X 100, etched in 25% HNO_3)**

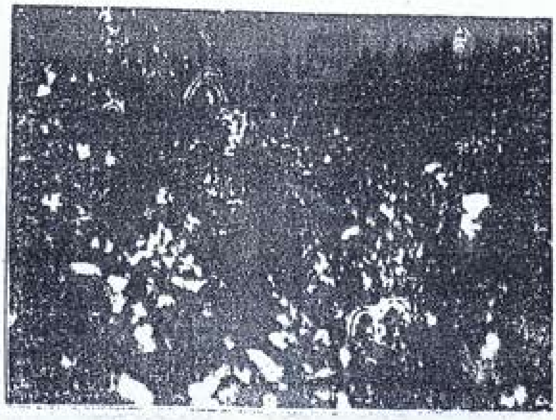

Plate A: Sample compacted at 96 Mpa (X 100, etched in 25% HNO_3)

Plate B: Sample compacted at 192 Mpa (X 100, etched in 25% HNO_3)