UNIVERSITI PUTRA MALAYSIA

FRACTURE TOUGHNESS OF IRON AND COPPER POWDER COMPACTS USING MODIFIED DIAMETRICAL COMPRESSION TEST TECHNIQUE

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FK 2018 131

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By

ALABI ABDULMUMIN AKOREDELEY

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Doctor of Philosophy

May 2018

DEDICATION

This thesis is dedicated to my beloved mother for all the love, prayers, trust, sacrifice and self-deprivation that she put herself through just to see her children grow into responsible adults. Hajiya Hafsat, you deserve more than a PhD, but I hope you will accept the dedication of this thesis, a product of 4-year struggle, as my little way saying that I will ever be grateful to you.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy.

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May 2018

Chairperson: Suraya Mohd Tahir, PhD
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ABSTRACT

In the industries today, metal components are increasingly being produced by powder metallurgy (PM) method. The PM method is highly efficient in cost of production and materials usage. However, PM components suffer from inhomogeneous density variation and are more likely to have internal cracks. These deficiencies usually make PM components prone to sudden fracture failure. A parameter that is known to define the rate at which cracks grow in a material is fracture toughness. Unfortunately, the fracture toughness of most metal powder compacts have not been determined due to lack of suitable test technique. This study developed a notching device that has the capability to provide uniform notches on the surfaces of powder compacts. The effectiveness of the notching device enhanced the determination of mode I fracture toughness (K_{IC}) of two metal powder compacts; iron and copper. A method known as the modified diametrical compression test technique (MDCTT) was also developed to measure the mode II fracture toughness (K_{IIC}) of the powder compacts. Finally, the study examined the influence of density on the rate of crack propagation in the compacts and developed mathematical relation that predicts fracture toughness from the relative density of either the iron or copper powder compacts. Notched samples of two types of metal powder; Hoaganas ASC100.29 iron powder and pure copper powder were prepared by uniaxial compaction in a rigid die using universal testing machine. The relative density of the powder compacts was determined as a fraction of the density of the compact to their corresponding solid metal before the diametrical compression test was carried out for each sample. The behavior of the cracks around the tip of the notch was examined using scanning electron microscope (SEM). A new equation was developed to calculate the values of K_{IIC} from the MDCTT. The results of K_{IC} for the iron powder compacts showed close agreement with values mentioned in the literature. The K_{IC} values for copper powder compacts range from 0.32 to 0.58 MPa.m$^{0.5}$ while the K_{IIC} for the iron and copper powder compacts ranged from 0.30 to 0.57 MPa.m$^{0.5}$ and 0.28 to 0.59 MPa.m$^{0.5}$ respectively. The ratio K_{IIC}/K_{IC} for the iron and copper powder compacts from this study showed good agreement with the predicted values of 0.87 and 1.04 based on the maximum tangential stress (MTS) and the minimum strain energy density (SED) criteria respectively. The agreement implies

that the developed MDCTT is reliable and can be used to measure the K_{IIC} of other metal powder compacts. Furthermore, the results also show that the rate of crack extension reduced as the density of the powder compacts increases. A generalized mathematical expression that relates fracture toughness and relative density has been successfully developed. This relationship will be beneficial for further analysis of crack propagation within metal powder compact.

Abstrak tesis yang disampaikan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

KELIATAN RETAK PADATAN SERBUK BESI DAN KUPRUM MENGGUNAKAN TEKNIK UJIAN MAMPATAN LURUS YANG DIUBAH SUAI

Oleh

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ABSTRAK

Dalam industri hari ini, kaedah metalurgi serbuk semakin kerap digunakan dalam penghasilan komponen-komponen logam. Kaedah ini sangat efisien dalam kos pengeluaran dan penggunaan bahan. Walau bagaimanapun, komponen ini diketahui mempunyai taburan kepadatan tidak sekata dan lebih cenderung mempunyai keretakan dalaman. Kekurangan ini biasanya menyebabkan komponen metalurgi serbuk retak secara tiba-tiba. Parameter yang digunakan untuk menentukan kadar di mana retakan merambat dalam bahan adalah keliatan retak. Malangnya, keliatan retak kebanyakan komposit serbuk logam belum dapat ditentukan kerana kekurangan teknik yang sesuai. Kajian ini membangunkan alat takukan yang mempunyai keupayaan untuk memberikan takuk seragam pada permukaan padatan serbuk. Keberkesanan alat takukan meningkatkan penentuan keliatan retak mod I (K_{IC}) bagi dua padatan serbuk logam; besi dan tembaga. Kaedah yang dikenali sebagai teknik ujian mampatan lurus yang diubah suai (MDCTT) juga dibangunkan untuk mengukur keliatan retak mod II (K_{IIC}) bagi padatan serbuk. Akhir sekali, penyelidikan ini mengkaji pengaruh ketumpatan terhadap kadar perambatan retak padatan dan membangunkan hubungan matematik yang dapat meramalkan keliatan retak daripada ketumpatan relatif padatan serbuk besi atau tembaga. Sampel bertakuk bagi dua jenis serbuk logam; serbuk besi Hoaganas ASC100.29 dan serbuk tembaga tulen dihasilkan melalui pemadatan ekapaksi dalam acuan tegar dengan menggunakan mesin ujian sejagat. Ketumpatan relatif padatan serbuk logam ditentukan sebagai pecahan ketumpatan padatan terhadap ketumpatan logam pejal yang berkaitan, sebelum ujian mampatan lurus dijalankan ke atas setiap sampel. Tingkah laku retak di sekeliling kawasan hujung takuk diperiksa menggunakan mikroskop pengimbas elektron (SEM). Satu persamaan baru telah dibangunkan untuk mengira nilai K_{IIC} menggunakan kaedah MDCTT. Keputusan K_{IC} untuk padatan serbuk besi menunjukkan nilai yang dekat dengan nilai-nilai yang terdapat dalam kajian literatur. Nilai K_{IC} untuk padatan serbuk tembaga adalah di antara 0.32 sehingga 0.58MPa.$m^{0.5}$ manakala K_{IIC} untuk padatan serbuk besi dan tembaga adalah di antara 0.30 sehingga 0.57 MPa.$m^{0.5}$ dan 0.28 sehingga 0.59 MPa.$m^{0.5}$ masing-masing. Nisbah K_{IIC}/K_{IC} untuk padatan serbuk besi dan serbuk

tembaga menunjukkan nilai yang dekat dengan nilai yang dijangkakan iaitu 0.87 dan 1.04 berdasarkan kriteria tangen tegasan maksimum (MTS) dan kriteria kepadatan tenaga terikan minimum (SED) masing-masing. Hubungan antara hasil eksperimen dan kriteria retakan membuktikan teknik ujian mampatan lurus yang diubah suai (MDCTT) boleh dipercayai dan boleh digunakan untuk mengukur K_{IIC} bagi serbuk logam lain. Selain daripada itu, keputusan juga menunjukkan bahawa kadar perambatan retak boleh menjadi perlahan dengan meningkatkan ketumpatan serbuk logam. Hubungan matematik am di antara keliatan retak dan ketumpatan relatif telah berjaya dihasilkan. Hubungan ini akan bermanfaat untuk analisa lanjut mengenai perambatan retak dalam padatan serbuk logam.

ACKNOWLEDGEMENTS

I wish to express my profound gratitude and sincere appreciation to my supervisor, Dr. Suraya Mohd Tahir for the guidance, encouragement, immeasurable contribution and total commitment to the success of this study. I am also highly indebted to members of my supervisory committee, Associate Professor Dr. Nur Ismarrubie Zahari and Associate Professor Dr. Azmah Hanim Mohamed Ariff for their treasured contributions, objective criticism and valuable advice.

I would like to thank the technical staff in the Department of Mechanical Engineering, Institute of Bioscience (IBS), and Institute of Advanced Technology (ITMA) for their assistance while using their facilities. To my colleagues at the Faculty of Engineering, I will remain grateful for all the useful advice and discussions.

I am also highly indebted to the authority of Ahmadu Bello University for sponsoring my study under the Assessment Intervention Fund "NEEDS 2014". To Professor Muhammad Dauda and Professor Terver Ause, assistance and encouragement.

To my friends and the members of the Alabi family, I am most grateful for your encouragement and financial support at critical times.

Finally to my wives and children, I seek your forgiveness for the emotional and psychological trauma, additional burden of responsibilities and unquantifiable hardship that my absence caused all of you.

I certify that a Thesis Examination Committee has met on 24 May 2018 to conduct the final examination of Alabi Abdulmumin Akoredeley on his thesis entitled "Fracture Toughness of Iron and Copper Powder Compacts Using Modified Diametrical Compression Test Technique" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS

a	Half the length of a crack
A_1	Active or fractured surface of TCDCTT compact
A_2	Active or fractured surface of MDCTT compact
c	Depth of notch or crack
TCDCTT	Through-cu diametrical compression test technique
CP	Compaction pressure
D	Diameter of sample
h	Rise or height of the cut out portion of the MDCTT sample
ISRM	International Society for Rock Mechanics
K_I	Mode I stress intensity factor
K_{IC}	Mode I fracture toughness
K_{II}	Mode II stress intensity factor
K_{IIC}	Mode II fracture toughness
MDCTT	Modified diametrical compression test technique
SED	Minimum strain energy density criterion
MTS	Maximum tangential stress criterion
N_I	Normalized stress intensity factor for mode I loading using the close-cracked disk
N_{II}	Normalized stress intensity factor for mode II loading using the close-cracked disk
N_{II*}	Normalized stress intensity factor for MDCTT compacts
P	Diametrical compression point load
P_1	Fracture load for TCDCTT compacts
P_2	Fracture load for MDCTT compacts
PM	Powder Metallurgy
R	Radius of a cylindrical sample, compact or disk
ρ_r	Relative density
t	Thickness of sample
t_1	Thickness of TCDCTT compact
t_2	Thickness of MDCTT compact
UTM	Universal testing machine
W	Half the width of the notch or crack
XSA	Cross section area
A	Crack angle (angle between the crack and the y-axis)
β	R/h
γ	w/h
σ	Tensile stress

σ_x	Tensile stress in the x-direction
σ_y	Tensile stress in the y-direction
τ_{xy}	Shear stress in the xy-plane

CHAPTER 1

INTRODUCTION

Powder metallurgy (PM) is a field of engineering that studies powder production and their useful applications. It has also been described as a forming process that involves consolidation of powdered materials into regular or irregular shaped components for use in different spheres of life. The forming process can be performed with hot or cold powders. When the powder consolidation is done in the cold state the compacted body is referred to as green body or green compact. PM process has been used to produce components from different powdered materials, but iron powder remains the oldest and the most used of them all. Other areas where the PM technique has found wide application include the ceramics and pharmaceutical industries, and for the production of composite materials. The main advantage of PM over other forming processes such as casting and forging is its cost effectiveness. PM process consolidates powders at temperatures lower than their melting points. It turns out products which are very close to their designed shape and dimensions, thereby eliminating the need for machining. It uses up over 95% of the starting powder leaving little or no scraps (Krar and Gill, 2003; Boljanovic, 2009).

1.1 Background of the Study

Some of the advantages which powder metallurgy (PM) has over other manufacturing techniques such as casting and forging are low production cost, higher energy efficiency and its ability to combine materials that are known to be incompatible. These virtues have made PM attractive for processing conventional and advanced materials. PM is a forming operation where one or more dry powdered material(s) is caused to fuse into a desired shape by the application of pressure. The consolidation is usually done in a rigid or flexible die using cold or preheated powder. Uniaxial compaction of dry powder in a rigid column-like die is the most used PM technique due to its simplicity. A fused powder ejected from a die is known as a green compact. Green compacts are usually heat treated to improve the physical and mechanical properties. This heat treatment process is known as sintering.

It is almost impossible to produce a flawless component irrespective of how advanced the manufacturing technique used may be. PM components are products of a well-established sequence of events which include powder mixing, powder transfer, compaction, ejection and post-ejection handling. Defects or cracks can form in a compact at any stage in this sequence, but they are more likely during the compaction and ejection stages (Jonsén et al., 2007). During the onset of compaction, weak inter-particle bonds are formed. The weak bonds are broken and give way for stronger bonds as the compaction progresses. The complex nature of powder compression makes it difficult to predict and stop compaction at the exact point where all broken bonds have transformed to stronger ones. During the ejection process, a green compact held to the walls of the die by radial forces is forcibly ejected. This action usually creates cracks in the green body (Jonsén, 2006). The degree of cracks is significantly reduced in compacts with high green strength. Defect due to cracks is known to be the leading cause of fracture failure in metal PM components. Another cause of cracks in PM components is wide inhomogeneous density variation (Zhou et al., 2017; Jonsén, 2006). Some of the factors that make it difficult to produce PM components with uniform density variation include the high flow of powder particles during compaction (Jonsén, 2006), friction between die walls and green compacts (Gethin et al., 2008; Zhou et al., 2017; Staf et al., 2017; Hjalmar et al., 2018), kinematics of the compaction, that is, the movement and the interactions between moving parts during compaction and ejection processes (Gethin et al., 2008; Enneti et al., 2013; Anbalagan et al., 2017).

1.1 Problem Statement

The need to reduce the cost of production, save energy, and produce materials with superior mechanical properties, has led to a tremendous increase in the volume of research work in material science and engineering. Components from iron and steels, the most used engineering materials, are usually produced by forging or casting processes. These processes require huge capital to set up, also consume an enormous amount of energy in production. Products of forging and casting processes usually have high inertia, and their properties can only be altered during production by alloying (another expensive technique, which usually requires a different set up), and after production by heat treatment (a highly technical and energy consuming technique). Metal powder compact is a class of material that has competed favorably with traditional iron and steels, especially in the automotive industry. In the automotive industry, metal powders have been used to produce parts such as self-lubricating bearings, oil pump rotors, gears, value seats and pulleys (Ramakrishnan, 2013; Erdem, 2017) Metal powder components are light-weighted and, require lesser capital investment and energy for production.

Metal powders have complex characteristics which make it difficult to predict their behavior during compaction. These features, in addition to cost and time, involve in conducting laboratory experiments, have made researchers dwell more on using computer software to simulate the behavior of metal powders compacts. As attractive as most finite element simulation methods are, the accuracy of their results depend on knowledge and reliability of available material parameter (Chtourou et al., 2002). In 2010, Tahir et al., used existing experimental data as input data while simulating the fracture toughness of iron compact. Jonsén and Häggblad (2007) validated the results of their model on the residual stress state of green metal compacts using existing experimental values (Jonsén and Häggblad, 2005). Obviously, it is essential to have more experimental studies into the properties of metal powders to provide ample data to enhance the accuracy of simulated results. Unfortunately, only a few experimental works have studied the mode I fracture toughness of metal powder compacts while none has been dedicated to its mode II. It is not sufficient to argue that the mode II fracture toughness of iron powder compact has not been given much attention because the growth of a crack in any mode of fracture begins with pure opening (Tahir and Ariffin, 2006a), because the mode II fracture toughness for virtually all other materials have been studied (Ayatollahi and Aliha, 2005; Jamali et al., 2015; Aliha and Rezaei, 2011; Backers and Stephansson, 2012a; Wang et al., 2016; Refat et al., 2005; Ayatollahi and Aliha, 2006; Aliha and Ayatollahi, 2008; Aliha et al., 2009). A review of the methods that have been used to study mode II fracture toughness shows that all of them have one or more critical requirement that can hardly be fulfilled by metal powder compacts. The most common of these requirements are the specified specimen geometry and size, the need to machine the test specimen, and post-formation handling.

Some of the identified gaps which this research has filled include:

1. There is the need to have a simple and inexpensive means of notching iron and copper powder compacts for diametrical compression test. The notching device should be able to produce notch of same dimension and geometry on different test specimens with high precision, and with little or no adverse effect on the properties of the bonded metal powder.
2. The experimental value of mode II fracture toughness for iron powder compact or any other metal powder compacts have not been reported.
3. The existing test methods for the determination of mode II fracture toughness cannot accommodate the peculiar nature of metal powder compacts. Hence, there is a need to find an alternative method.

1.2 Objectives

The objectives of this study are:

1. To develop a notching device that enhances the diametrical compression test technique for the determination of mode I fracture toughness of iron and copper powder compacts.
2. To develop a test method for the determination of mode II fracture toughness of iron and copper powder compacts.
3. To develop the relationship between fracture toughness and density of iron and copper powder compacts.

1.3 Scope of Study

This research work is limited to an experimental determination of the mode I and mode II fracture toughness of iron (Hoaganas ASC100.29) and copper powder compacts using the concept of the diametrical compression technique. A notching device was produced. The notching device enabled the powder samples to be compacted and notched simultaneously in a rigid die during a uniaxial compaction technique called “the integrated compaction and notching method”. A new test technique was developed for the determination of the mode II fracture toughness of iron and copper powder compacts. The technique is known as the modified diametrical compression test technique (MDCTT). A new equation was developed for evaluating the mode II fracture toughness of iron and copper powder compacts using the MDCTT. The results of the fracture toughness obtained from the use of the notching device and the MDCTT were validated by comparison with the theoretical predictions from two fracture criteria; the maximum tangential stress (MTS) criterion and the minimum strain energy density (SED) criterion. A new mathematical relation was also proposed to describe the influence of relative density of iron and copper powder compacts on their fracture toughness. The properties of the two metal powders were studied under four compaction pressures. These pressures are 206.87, 238.70, 270.53 and 302.36MPa.

1.4 Thesis Overview

The thesis is structured to present this research work in five chapters.

Chapter 1 gave a general introduction to powder metallurgy (PM) as a production process, its advantages, and shortfalls. The problem statements, objectives and scope of the research were highlighted.

Chapter 2 began with an explanation of terms relevant to understanding metal powder compaction. The chapter also presented a review of studies relating to fracture toughness as a material property, methods of measuring fracture toughness, fracture criteria and the relationship between metal powder compact density and compaction pressure, green strength and fracture toughness. Chapter 2 was concluded by presenting a summary which justified the need to develop a notching device and to enhance the technique for the determination of mode I fracture toughness of metal powder compacts. The summary also justified the need to develop a new method for measuring the mode II fracture toughness of metal powder compacts, and the need to study the influence of compact density on the fracture propagation in metal powder compacts.

Chapter 3 discussed the materials studied and the methods used to achieve the set objectives of the study. The chapter presented a detailed explanation of the production of the through-cut diametrical compression technique (TCDCTT) samples used for the determination of mode I fracture toughness for metal powder and the development of a test method known as modified diametrical compression technique (MDCTT) for measuring the mode II fracture toughness of metal powder compacts. The chapter also presented the development of a new equation for the determination of the mode II fracture toughness of iron and copper powder compacts produced by the modified diametrical compression test technique (MDCTT). The design of a device for notching metal powder compacts during the compaction process (integrated compaction-notching method) was also reported. This device eliminates the need for machining the consolidated metal powder, and it replicates the notch with accuracy and precision. The chapter ended with the discussion on the measurement of density and the morphology of fractured surfaces of the metal powder compacts.

Chapter 4 presented and discussed the results of the findings of this study. The mass and thickness of the TCDCTT metal powder compacts were presented. The influence of compaction pressure on compact thickness, fracture loads and mode I fracture toughness were discussed. The data obtained from the compaction and compression of the developed MDCTT samples was also presented. The mode II fracture toughness for the iron and copper powder compacts were determined from the developed equation. The reliability of the results obtained from the use of the newly developed MDCTT was validated using the maximum tangential stress (MTS) and the minimum strain energy density (SED) criteria. The chapter also presented the findings on the influence of the densities of iron and copper powder compacts on their mode I and mode II fracture toughness. An improved relative density in the compacts of the iron and copper powders was found to slow down the rate of crack propagation. Morphological study was presented to support the assertion that improved that relative density and fracture toughness are directly related. Finally, a generalized equation was developed that described the relationship between the density of iron and copper powder compacts and their fracture toughness. The generalized equation contained some constants that were related to the thermal conductivity of iron and copper metals.

Chapter five highlighted the conclusions drawn from this study and also the suggested recommendations for future research works.

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