

UNIVERSITI PUTRA MALAYSIA

MECHANICAL PROPERTIES OF ULTRA-FINE GRAIN ALUMINIUM AL6063 AND PURE COPPER PROCESSED BY EQUAL CHANNEL ANGULAR EXTRUSION METHOD

JAMSHID NEMATI

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

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DEDICATIONS

Dedicated to my family and in particular to my wife, *Tayebeh* whose love, support, and encouragement are the most wonderful of the many blessings that God has granted me.



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirements for Degree of Doctor of Philosophy

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By

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July 2015

Chairman: Professor Shamsuddin Bin Sulaiman, PhD Faculty : Engineering

Severe plastic deformation (SPD) is one of the processes used to refine the microstructure of materials among which the Equal Channel Angular Extrusion (ECAE) is the most common method. ECAE has gained much interest over the past decades. In this study, pure copper and aluminum alloy 6063 grain refinement were performed using the ECAE method. The materials were extruded up to eight and six passes at room and 200°C temperatures with a constant ram speed of 20 and 30 mm/min, respectively, following route A through a die angle of 90° between the die channels. Optical microscopic examinations revealed pure copper grain refinements in the range of 32 µm to 4 µm after eight passes whereas for Al-6063 alloy the grain diameter reduced from 45 µm to 2.8 µm after six passes of ECAE. The extruded specimens were tested under quasi-static, medium and high strain rate loadings using various testing machines. Mechanical properties of the extruded material were obtained at different strain rates. For the pure copper, it was found that the maximum increased of ultimate strength of 80% occurred after the second pass for the V=200 mm/min. The total increase of ultimate strength after eight passes was around 100%. Furthermore, hardness, increased to a maximum of 36% after eight passes.

The results of Al-6063 tensile tests indicated that the tensile yield stress (YS) and ultimate tensile strength (UTS) of the extruded specimens increased significantly after 5 passes of ECAE process. The average increase was found to be around 70%, regardless of the tension velocities. The hardness measurements were made on different locations of the billet. For Al-6063, the results indicated around 90% increase in microhardness after 5 passes. The results for pure copper illustrated that the most increase of hardness (about 18%) occurs after the 2^{nd} pass. A total of around 36% increase in hardness was observed after 8 passes.

A maximum increase in impact energy absorption of 100% was achieved from Charpy tests after eight passes and 90% after six passes of the ECAE process for pure copper and Al-6063 alloy, respectively. In addition, bending fatigue test results indicates that for fatigue tested pure copper specimens, the results of fatigue tests indicated that a significant improvement in fatigue life occurred after the 2nd pass. For low stresses, a



maximum increase in fatigue resistance of approximately 500% was observed. The results of fatigue tested Al-6063 specimens indicate that major improvements in fatigue resistance occurred after the first pass. The impact strength of extruded specimens was also evaluated for different passes at a strain rate of 1800 s⁻¹ using Split-Hopkinson pressure bar (SPHB). The results indicated that the major strength improvement for Al-6063 and pure copper in the 5th and 6th passes and 6th and 8thoccurred, respectively. Finally, ECAE process was simulated using the DEFORM-3D software through a three-dimensional analysis.



Abstrakt tesis yang dikemukakan kepada Senat Universiti Pura Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

SIFAT MEKANIKAL BUTIRAN HALUS ALUMINIUM AL6063 DAN TEMBAGA TULEN YANG DIPROSES DENGAN KAEDAH SEMPERITAN SALURAN SAMA PENYEMPERITAN SUDUT (ECAE)

Oleh

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Mengubah bentuk plastik teruk adalah satu proses yang digunakan untuk mengecilkan struktur mikro bahan dimana antara cara yang digunakan ialah Saluran Sama Penyemperitan Sudut (ECAE). ECAE telah diminati beberapa dekad lalu. Dalam kajian ini, tembaga tulen dan aloi Aluminium 6063 penghalusan bijian/struktur telah dilakukan menggunakan kaedah ECAE. Bahan-bahan ini telah disemperitkan sehingga lapan dan enam laluan pada suhu bilik dan 200°C dengan halaju ram yang tetap 20 dan 30 mm/min, melalui sudut acuan 90° antara saluran-saluran acuan. Pemeriksaan mikroskop optik mendapati penghalusan bijian tembaga tulen adalah antara 32µm hingga 4µm selepas melalui lapan laluan tetapi untuk aloi Al-6063 garispusat bijian berkurang dari 45µm ke 2.8 µm selepas enam laluan ECAE. Sifat-sifat mekanikal hasil ECAE telah dikaji. Spesimen semperitan telah diuji dibawah kuasi-statik, bebanan kadar terikan pertengahan dan tinggi menggunakan berbagai mesin ujian. Sifat-sifat mekanikal bahan tersemperit diperolehi pada kadar terikan berbeza. Untuk tembaga tulen, didapati kekuatan muktamad maksima 80% bertambah berlaku pada laluan kedua dengan kelajuan V=200 mm/min. Jumlah kenaikan kekuatan muktamad selepas laluan kelapan adalah 100%. Tambahan pula, kekerasan bertambah sehingga maksima 36% selepas lapan laluan. Specimen secara fractografi menunjukkan bahawa mekanisma keretakan adalah tidak bergantung kepda kadar terikan.

Keputusan ujian tegangan Al-6063 menunjukkan tegasan alah tegangan (YS) dan kekuatan tegangan muktamad (UTS) spesimen disemperit bertambah dengan ketara selepas 5 laluan proses ECAE. Purata kenaikan sekitar 70% telah diperolehi tanpa mengira kelajuan tegangan. Ini menunjukkan sifat tegangan specimen tidak dipengaruhi oleh kadar terikan.Pengukuran kekerasan telah dibuat pada kedudukan berbeza bilet. Untuk Al-6063, keputusan menunjukkan penambahan 90% kekerasan mikro selepas 5 laluan. Keputusan untuk tembaga tulen menyatakan kekerasan banyak bertambah (18%) berlaku selepas laluan kedua. Jumlah sebanyak 36% pertambahan kekerasan dilihat selepas 8 laluan. Penambahan maksima penyerapan tenaga hentaman 100% dicapai melalui ujian Charpy selepas lapan laluan dan 90% selepas enam laluan proses ECAE untuk tembaga tulen dan Al-6063.



Sebagai tambahan, specimen lesu bentuk-tulang-anjing telah dihasilkan dari spesimen semperitan dengan laluan dan tanpa laluan. Untuk ujian kelesuan specimen tembaga tulen, keputusan kelesuan menunjukkan penambahbaikan ketara kehidupan keletihan berlaku selepas laluan ke 2. Untuk tegasan rendah, didapati tahan keletihan bertambah maksima hampir 500%. Keputusan ujian kelesuan spesimen AL-6063 menunjukan penambahbaikkan major dalam tahan kelesuan berlaku selepas laluan pertama. Kekuatan hentaman specimen semperitan juga dinilai pada laluan berbeza dengan kadar terikan 1800 s⁻¹ menggunakan Split-Hopkinson Pressure Bar (SPHB). Keputusan menunjukkan penambahbaikan kekuatan major untuk Al-6063 dan tembaga tulen dalam laluan ke 5 dan ke 6, dan laluan ke 6 dan ke 8. Akhirnya, proses ECAE telah disimulasikan menggunakan perisian DEFORM-3D melalui analisis tiga-dimensi.



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I certify that a Thesis Examination Committee has met on 13 July 2015 to conduct the final examination of Jamshid Nemati on his thesis entitled "Mechanical Properties of Ultra-Fine Grain Aluminium AL6063 and Pure Copper Processed by Equal Channel Angular Extrusion Method" 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

ARB	Accumulative Roll Bonding
ASTM	American Society for Testing and Materials
CEC	Cyclic Extrusion-Compression
CVD	Chemical Vapor Deposition
d	Average grain diameter
ECAE	Equal Channel Angular Extrusion
EDM	Electro Discharge Machining
FEM	Finite Element Method
HCF	High-Cycle Fatigue
HPT	High Pressure Torsion
IGC	Inert Gas Condensation
K _v	Material constants
LĊF	Low-Cycle Fatigue
MF	Multiaxial Forging
NC	Noncrystalline Materials
OM	Optical Microscope
PEEQ	Equivalent Plastic Strain
RCS	Repeated Corrugation and Straightening
SEM	Scanning Electron Microscope
SHPB	Split-Hopkinson Pressure Bar
SPD	Severe Plastic Deformation
UFG	Ultrafine Grained Materials
UTS	Ultimate Tensile Strength
VH	Vickers Hardness
YS	Yield Strength
ó6	Strain rate
μ	Friction Coefficient
1.	Material constants
у	Yield stress
	Die channel angle
*********************************	The angle of the arc of curvature of die

CHAPTER ONE

INTRODUCTION

1.1. Background of the Study

In materials engineering, it is essentially important to characterize and improve the mechanical properties, among which the strength, ductility, fatigue life, and others. The fact is that microstructure, which affects the material mechanical performance, also determines the mechankecn" rtqrgtvkgu"qh"cp{" o cvgtkcnu" fktgevn{0" J gpeg."vjg" o cvgtkcnu" average grain size, particularly plays a very substantial and dominating role. In this context, those materials which have grain sizes ranging from 100 to 1000 nm, i.e. <1-

m, are called Ultra-Fine Grain (UFG) materials (Srinivasan, et al., 2006), which even have grain sizes bigger than the nano-materials and are seemingly recognized as the materials having grain sizes less than 100 nm. While there have been voluminous attentions given to the bulk UFG materials throughout the last two decades, several techniques have also been simultaneously devised aimed at manufacturing UFG ocvgtkcnu0"Hqt"kpuvcpeg."kp"qtfgt"vq"vtcpuhqto"cpf"tghkpg"vjg" ocvgtkcnu0" oketquvtwevwtg." the Severe Plastic Deformation (SPD) process is exploited, which is in detail describes as a kind of deformation being able to deform a billet of material beyond a true strain of 4.00. In addition, in order to refine the microstructural grain, numerous kinds of SPD are commonly utilized. Illustrations of such kinds are drawing, rolling, swaging, 3- axes deformation, and equal channel angular extrusion (Heffner, 2008).

A lot of of endeavors have been so far borne directed on manufacturing the bulk ultrafine/nanostructured materials directly from micro structured ones by making use of the SPD. Particularly with the equal channel angular extrusion/processing (ECAE/ECAP) which is referred to as the most economical method and the easiest one to execute among the other SPD methods. It is admitted that one reason for such properties is the simplicity in both processing and tooling (Abd El Aal, 2011). The first instance of its development was reported in the former Soviet Union in the early 1990s by V.M. Segal (Segal, 1995). Since then, the materials community has tremendously focused on the ECAE process on account of its capability to yield ultrafine- grained metals as well as offering novel properties (Chen, et al., 2003).

It is accentuated that the ECAE process was principally concomitant with structure refinement to sub-micron scale (Segal, 2004). Refining the grain size leads to the dislocation pile-up at the grain boundary which accordingly hampers the dislocation oqxg ogpv"cu" ygnn"cu"dquvkpi"vjg" ocvgtkcnøu" {kgnf"uvtguu0 It is repeatedly proved that tjku" vgejpkswg" ecp" dg" wugf" hqt" ukipkhkecpvn{" gpjcpekpi" vjg" ocvgtkcnøø" ogejcpkecn" properties in consort with their microscopic structures.

A review was undertaken to examine using the ECAP for grain refinement by altering the conventional ECAP with the purpose of boosting the process efficiency and the techniques used for up-scaling the procedure as well as processing the materials which were hard-to-deform (Valiev and Langdon, 2006). Figure 1.1 schematically exhibits the ECAE process wherein the specimens with square or circular cross section are strained by consecutive extrusion processes.

 \bigcirc



Figure 1-1:Schematic process of ECAE (Salem, H. G., & Lyons, J. S. 2002)

The ECAE technique revolves around refining the metals and alloys microstructure which ultimately results in the material strengthening. According to the Hall-Petch relationship (Etch, 1953), this technique is mathematically described as follows:

(1.1)

where $_y$ is the yield stress, $_o$ and K_y ctg" vjg" o cvgtkcnuø" eqpuvepvu." y jkng" *d* is the average grain diameter. The ECAE is particularly distinctive due to the fact that the cross sectional area of the workpiece will be unaffected in the course severe deformation; nonetheless, extrusion and drawing the area decreases in other processes such as forging. It should be noted that the material in the ECAE technique is extruded through a channel and around a corner which have mostly an angle of 90°. Once the work piece passes by this corner, it experiences fairly large strains, at times, of the order of 1.5. Furthermore, the strains at the consecutive passes amass causing more material refinement as the section of the workpiece will be equal on the entrance and exit of the die.

It should be pinpointed that better enhancement will be given into the billet mechanical behavior so long as the number of passes increases (up to 7 to 8 times of extrusions). The ECAE is advantageous as the billet is capable of supporting severe deformations as well as sustaining large strains larger than one while it never struggle with the change in the dimensions. The core fact is that the materials grain turns out to be finer owing to these large strains. It should be noticed that the grain refinement is carried on after each extrusion pass. This process can eventually yield a nano-structures material (Patil Basavaraj, et al., 2008) while such a process is not burdened with the weaknesses typically associated the other producing techniques like the milling and compaction. In details, the latter mentioned techniques are concomitant with few issues, including impurities, cavities, buckling and fracturing.

Over the last two decades, a great deal of research has been conducted to comprehensively investigate the microstructural aspects of the materials which have been generated through the ECAE. The microstructure of a copper was scrutinized by Huang et al. (2004) which had been developed by means of the ECAE to a strain ý 8 employing two different strain paths by way of rotating the billets through 90 °or180 ° between each pass. Moreover, the nano-structured cqrrgtøu" *PE+" rtqrgtvkgu" ygtg" significantly affected by the heat treatment and the hardening cyclic deformation in the viscoplastic regime (Kommel et al., 2007). Also, the mechanical properties related to a 6063-T1 aluminum alloy have been studied while this alloy was processed via the ECAP method up to nine passes, revealing that the SPD (ECAP processing) could significcpvn{" gpjcpeg" vjg" Cn" 8285" cmq{uø" ogejcpkecn" rtqrgtvkgu" vjtqwij" itckp" refinement (Serban et al., 2012). Furthermore, it is found that at the room temperature, the annealing temperature can remarkably influence the microhardness of the asprocessed material (Daly, et al., 2009).

Moreover, under constant stress testing, a significant enhancement of fatigue limit and fatigue life has been reported in the ultrafine-grained state revealing that numerous cyclic properties related to the severely-predeformed materials having fine grains can be reorganized with regard to the HallóPetch grain boundary hardening as well as dislocation hardening (Vinogradov, et al., 2001). Likewise, the tensile properties and impact toughness of Znó40Al alloy subjected to the ECAE have been examined demonstrating that elongation to fracture noticeably upsurges corresponding to the rise in the number of ECAE passes as well as proving that there is an escalation in the strength after the first pass while there will be a decrease for the succeeding passes. Such softening is claimed to occur by reason of the deformation-induced homogenization alongside the incessant change in the composition of the constituting phases with the number of passes. Another related conclusion is that as a result of the significant growth in ductility, multi-pass ECAE could be employed to improve the impact toughness of the alloy (Pureck et al., 2004).

1.2. Statement of the Problem

As stated earlier, scientists and engineers have been fascinated in ultrafine grains (UFG) and nanostructured (NC) materials for more than two decades. In recent times, many advances have also been obtained in processing and characterizing these two materials due to their boosted properties, namely their high tensile strength, high toughness, excellent fatigue life, as well as their likelihood to get superplasticity at the low temperatures (Kim et al., 2003; Tham et al.,2007). Along with an ever-increasing demand on the improved materials performance, there is an urge for ultra-fine grainsized materials which yield greater strength while there is no deterioration in the o cvgtkcnuø" fwevknkv{0" Qh" pw ogtqwu" gzkuvkpi" ugxgtg" rncuvke" fghqt o cvkqpu." vjg" GECG" technique has proved capable in developing the ultra-fine grained microstructures in bulk samples. It is also substantiated by Segal that the simple shear (SS) taking place on the die intersection plane can be used to estimate the deformation in the ECAE under ideal conditions (Beyerlein et al., 2008).

To date, it is discerned that the majority of the published literature investigating the effect of the ECAE process has only examined few mechanical properties and/or microstructure of the materials under study, leaving out a dearth of much more comprehensive studies. Moreover, there are still some unanswered questions on the

impact this rtqeguu"korqugu"qp"vjg" ocvgtkcnuø" ogejcpkecn"rtqrgtvkgu0" Y jcv"hqnnqyu"ku"c" brief account of such questions:

- **1.** Is there any limitation in grain refinement in a metal or alloy using the ECAE method?
- **2.** Does the improvement of mechanical properties of the material keep rising with grain refinement? Or does it have a critical point?
- 3. Vq" y j cv" gzvgpv" ecp" v j g" k o r tqxg o gpv" qh" o cvgtkcnuø" o ge j cpkecn" r tq r gtvkgu" dg" achieved using the ECAE method?
- **4.** Are the responses if the ECAEed materials in quasi static, dynamic, and cyclic loads similar to the normal materials?
- **5.** What is the fracture mechanism regime of the ECAEed materials under different loading conditions?

To address the above mentioned questions, this study was conducted to investigate the mechanical properties and microstructure as well as their relationship of pure copper and aluminum alloy 6063 processed using the multi-pass ECAE method.

1.3. Research Objectives

The main objective established in this research study was to refine and homogenize the microstructure of commercially pure copper and aluminum alloy 6063 via the Severe Plastic Deformation (SPD) employing the Equal Channel Angular Extrusion (ECAE). Moreover, the effects of grain refinement on mechanical properties improvement of the processed materials using ECAE were examined. To fulfill the main objective, the following specific objectives were addressed:

- 1. To predict the force and speed of the plunger during the ECAE process.
- 2. To determine the strength, structure and fracture toughness of extruded pure copper and Al-6063 alloy after different ECAE passes.
- 3. To determine the fatigue life of the extruded materials.

1.4. Scope and Limitations of the Study

The focus of this research is to study the mechanical properties improvements while it deals with grain refinement of the materials by SPD is employing the ECAE method and explores its effects on microstructure and mechanical properties. In details, the study focused on AL-6063 alloy and pure copper as they both entailed satisfactory workability as well as possessing extensive applications in industry. The research was conducted through the two subsequent stages while only one of the materials was examined in each stage:

C During the first stage, the pure copper was employed and the received material was annealed and extruded through the ECAE method for a maximum of eight passes. The point is that this process was carried out at the room temperature in a die made up of two channels of equal cross-section intersecting at an angle 90°, having corner angle of 20°. Afterwards, by using 2, 4, 6 and 8 passes, the ECAE tests were executed while eight specimens were tested during each pass. In order

to prepare the specimens for mechanical characterization and microstructural examination of the extruded materials, the tested billets were employed.

During the second stage, identical processes were executed for the annealed Al-6063 alloy. In details, the ECAE tests were achieved by employing 1, 2, 3, 4 and 5 passes following route A at the temperature which varied from 195°C to 205°C. Eight specimens were tested during each pass. The tested billets were used to prepare the specimens for mechanical characterization and microstructural examination of the extruded materials.

It should be asserted that this research study encountered two main limitations. In the first place, by increasing the number of ECAE passes, the extrusion force will increase, necessitating a pressing machine with higher capacity as well as requiring a die and plunger with high strength and such demands were almost impossible. In the second place, the extruded specimens became shorter after each pass of the ECAE process; this was the case because after a definite amount of the ECAE passes, their suitable length became too short as to prepare the samples for most of the examination tests on the mechanical properties; these tests included the tensile, fatigue, fracture toughness, and so on.

1.5. Organization of the Thesis

This thesis is organized in ten chapters. Chapter one presents a general introduction consisting; Background of the Study, problem statement, research objective and scope and Limitations of the Study. In chapter two a review of literature that relates to the ECAE process and its effect on the mechanical properties of processed materials as well as their microstructure is presented. Description of methods used in the study and investigated materials provided in chapter three.

Chapter four discusses finite element and metallurgical study of properties of deformed pure copper by ECAE at various strain rates. An investigation of microhardness characteristics of Al-6063 alloy processed by Equal Channel Angular Extrusion is presented in chapter five. Chapter six discusses the improvements in the microstructure and fatigue behavior of pure copper using equal channel angular extrusion (ECAE).

Chapter seven discusses the experimental study of impact strength of Al-6063 alloy processed by Equal Channel Angular Extrusion. Chapter eight is related to the results and discussion of the development of microstructure and fracture toughness of AL-6063 alloy using equal channel angular extrusion. Chapter nine discusses the effect of equal channel angular extrusion on Al-6063 bending fatigue characteristics.

Chapter ten discusses the outcomes of the research; provides answers to the research questions and also how the objectives of the research were fulfilled. The chapter ends with conclusions and setting a direction for future research which could further investigate some aspects of this research.

5

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