



**UNIVERSITI PUTRA MALAYSIA**

***PREPARATION AND CHARACTERIZATION OF NANO CRYSTALLINE  
STRUCTURES ON CK60 STEEL BY DRILLING METHOD***

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STRUCTURES ON CK60 STEEL BY DRILLING METHOD**

**By**

**SHOHREH NOSRATI**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,  
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Abstract of thesis presented to the Senate of Universiti Putra Malaysia, in fulfilment of the requirements for the degree of Master of Science

**PREPARATION AND CHARACTERIZATION OF NANO CRYSTALLINE  
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June 2013

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Drilling as a surface severe plastic deformation (SSPD) method has been applied to commercial CK60 steel plate to create a nano crystalline (NC) structured layer. In the present study, the CK60 steel plate has been quenched in room temperature water from 950 °C and tempered at 350°C for one hour.

Drilling was done with use of a Ti-oxide coated drilling bit at 5, 10, 15 and 20 m/min cutting speeds. The microstructure of the samples was studied by an Optical Microscope and by a field emission scanning electron microscope (FESEM). The formation of an NC layer having a grain size in the order of 50 nm was confirmed by SEM observation and by applying the Hall-Petch formula on the samples drilled at a 20 m/min speed. The fine grain zone created was separated from the base metal structure and clear boundary with a 1 to 10  $\mu\text{m}$  thickness when the drilling speed changed from 15 to 20 m/min.

The result of the micro-hardness test determined that the hardness of the surface NC layer increased by more than twice compared with the coarse grain structure of the base metal. The annealing test on the nano crystallized samples in the range 400 to 650 °C showed that the NC layer is thermally stable and the micro-hardness of the surface layer showed a small decrease after 1 hr tempering at 650 °C from 9.8 to 9.1 GPa.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai  
Memenuhi keperluan untuk ijazah Master Sains

**PENYEDIAAN DAN PENCIRIAN NANO STRUKTUR KRISTAL ON CK60  
STEEL OLEH KAEDAH PENGGERUDIAN**

Oleh

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Kaedah menggerudi sebagai salah satu kaedah ubah bentuk plastic keterlaluan pada permukaan (SSPD) telah digunakan ke atas plat keluli komersial CK60 bagi menghasilkan lapisan struktur nano kristal (NC). Di dalam kajian ini, plat keluli CK60 telah direndamkan di dalam air yang bersuhu bilik daripada suhu 950°C dan dilembutkan pada suhu 350°C selama 1 jam. Penggerudian telah dijalankan dengan menggunakan mata gerudi bersalut titanium oksida pada kelajuan 5, 10, 15 dan 20 m/min.

Mikrostruktur sampel telah dikaji dengan menggunakan mikroskop ringan dan mikroskop imbasan electron pancaran medan (FESEM). Pembentukan lapisan NC yang bersaiz butiran 50 nm telah disahkan melalui pemerhatian menggunakan mikroskop imbasan elektron (SEM) dan juga dengan menggunakan formula Hall-Pitch pada sampel yang telah digerudi pada kelajuan 20 m/min.

Zon berstruktur butiran halus yang telah dihasilkan terpisah daripada struktur logam asas dengan ketebalan sempadan daripada 1 hingga 10 mikron apabila kelajuan penggerudian diubah daripada 15 kepada 20 m/min.

Hasil ujian mikrokekerasan menunjukkan bahawa kekerasan lapisan permukaan NC meningkat lebih dua kali ganda berbanding dengan struktur butiran kasar logam asas. Ujian peneguhan ke atas sampel nano kristal pada suhu 400°C sehingga 650°C menunjukkan bahawa lapisan NC adalah stabil manakala ujian mikro kekerasan bagi lapisan permukaan hanya menunjukkan sedikit penurunan nilai selepas satu jam pelembutan pada suhu 650°C, iaitu daripada 9.8 GPa kepada 9.1 GPa.

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## APPROVAL

I certify that a Thesis Examination Committee has met on 29<sup>th</sup> of June, 2013 to conduct the final examination of Shohreh Nosrati on her thesis entitled **“PREPARATION AND CHARACTERIZATION OF NANO CRYSTALLINE STRUCTURES ON CK60 STEEL BY THE DRILLING METHOD”** in accordance with the Universities and University Colleges Act 1971 and the Constitution of Universiti Putra Malaysia [P.U. (A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science in Mechanical Engineering.

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## **DECLARATION**

I declare that this thesis is my original work except for the quotations and citations which have been duly acknowledged. I also declare that this thesis has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

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**SHOHREH NOSRATI**

Date: 28 June 2013

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## LIST OF ABBREVIATIONS

BCC	Base Cubic Centre Crystal
CGM	Coarse Grain Materials
FCC	Face Cubic Centre Crystal
FESEM	Field emission scanning electron microscope
FGM	Fine Grain Material
NC	Nano Crystalline
NM	Nano Material
NS	Nano Structure
OM	Optical Microscope
SPD	Severe Plastic Deformation Methods
SSPD	Surface Severe Plastic Deformation Methods
SEM	Scanning Electron Microscope
UFG	Ultra-Fine Grain structure

## **CHAPTER 1**

### **INTRODUCTION**

#### **Nano-crystallization of steels**

Over the past several decades, fine grain and more recently nano-crystalline materials have been deeply studied to make metallic parts with improved properties and durability. The steel industry has quite a long history in terms of human life and many attempts have been made to design new steels and alloys besides developing treatment processes to improve the mechanical and chemical properties of steel. Considering the fact that steel has the largest market and applications of all the metals, any treatment process which improves the abilities and properties of steel can be very important from the engineering and financial points of view.

Iron is the most widely used of all the metals and its production is almost 95 % of worldwide metal production. Iron and steel together have massive applications in the automobile and shipping industries, the construction of buildings and the fabrication of more sensitive engineering parts and components. Bearing in mind the importance of iron and steel in our daily life, any improvement in the properties of steel can spawn great engineering feats and provide added commercial value. In this regard, along with several matured techniques and process in the steel industry, scientists are still interested to work on novel refining processes such as new heat treatment cycles or controlled solidification processes (Symeonidis, 2008).

During the past 100 years, material scientists and engineers have designed a vast amount of special steel and ferro - based alloys to respond to the increasing demands of industry for new steel with higher mechanical, chemical and electrochemical properties. In addition to the attempts of engineers to create new steel with better properties, improvements in new crystalline structure modification and heat treatment techniques have also been considered as a more economical way to meet the new demands of engineering.

Grain refinement is one of the earliest techniques for improving the mechanical properties of steel. Controlling the grain size of steel by controlled solidification casting, pre or post heat treatment cycles, cold works and many other processes are based on the fact that decreasing the grain size improves the mechanical properties such as hardness and wear resistance. The common definition of this relationship is stated by the Hall-Petch formula (Vempaire, 2004) which shows a reverse relationship between the strength of steel and the grain size. Decreasing the grain size limits the free sliding space for dislocations and increases the required energy for deformation therefore enhancing the hardness. Moreover, grain refinement increases the grain boundary density and therefore increases the structural energy.

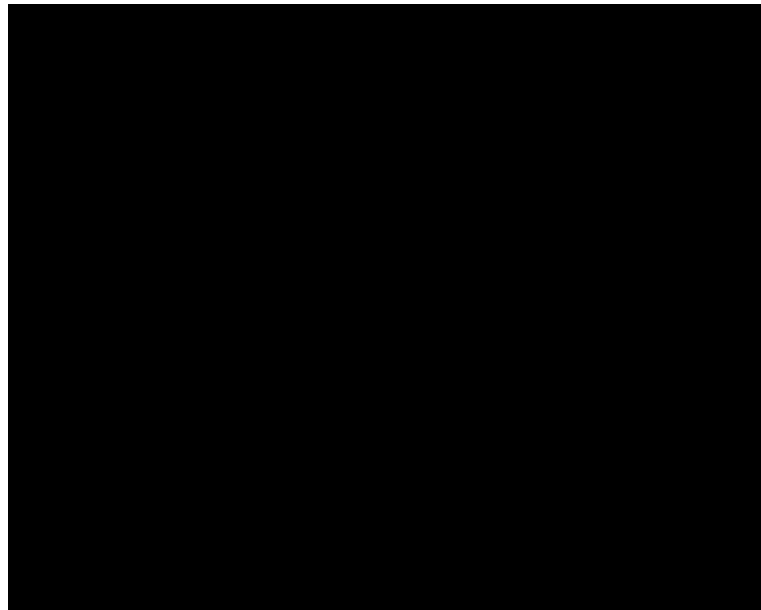
Metallurgists and materials engineers have devoted much effort into designing new grain refinement processes and techniques to produce ultrafine grain materials. Heat treatment is a matured science to control the crystalline structure and the grain size of metallic parts based on their phase diagrams, and it has vast applications in the steel industry today. Controlled solidification (Liu, 2000) in the metal casting process is a well-known technique to create fine grain structures, but it is a proven fact that grain refinement under cold work or mechanical force gives special properties to the metallic specimen (Vempaire, 2004). This effect is called cold work

hardening which has been discussed in many text books based on dislocation theories.

Recent achievements in nano crystallization processes and decreasing the grain size of steel to sub-micron sizes have created interesting properties such as a high level of hardness along with a suitable toughness. Many new nano-crystallization techniques and processes have been introduced and developed during the past decades such as using an ultrasonic frequency during solidification (Han, 2007). Meanwhile applying intensive mechanical deformation in order to break down grains seems more applicable, simpler and economical to create steel with a nano crystalline structure. Nano crystallization may help engineers to choose low alloy and carbon steels instead of special alloy steels to economically improve the life time of steel parts.

Creating a fine grain structure could have positive or negative effects on some properties such as corrosion resistance, depending on the working conditions of the final product. However, nano crystallization almost always improves the mechanical properties and hardness (Valiev, 2006; Vempaire, 2004, Nosrati, 2011). Based on this perception of developing the mechanical properties of steel, new grain refinement and nano crystallization techniques have been introduced in recent decades with the general title of Severe Plastic Deformation (SPD) methods such as sliding wear, ball milling (BM), high pressure torsion and shot peening (SP). As a simple description, applying a high deformation force to the crystalline structure creates a high density of dislocations and breaks the grains down to smaller sizes. If SPD techniques reach a certain level of strain rate a nano crystalline structure can be achieved. Although different SPD methods to create surface and bulk nano crystalline structures have been introduced and tested, to use these processes on an industrial scale and in real production situations it seems that much more research

work and basic data are required. More reliable and tabulated data are needed to be able to define theories and relationships for nano sized material, and then engineers will be able to safely apply these new parameters in their designs.



**Figure 1.1 Surface cracking failure of a steel pressure vessel with 15 cm thickness (Hayes, 1998)**

Considering the fact that most industrial part failures are initiated at the surface, tribological and surface properties are essential parameters in most steel applications. The surface properties under many conditions control the whole bulk steel part working life time. For example when we design a heavy pressure vessel, a simple surface crack created by fatigue may cause the total failure of the vessel (Figure 1.1). Moreover, in many severe mechanical working conditions such as the blades of a gas turbine (Figure 1.2), wear resistance and surface hardness are the main properties in design and a proper surface treatment or coating allows us to be able to fabricate

high quality and reliable steel parts by using cheaper steel instead of expensive high alloys or super alloys.

**Figure 1.2 Erosion on the blades of a gas turbine (ASI report, 2010)**

### **1.2 Nano-crystallization techniques**

Techniques such as a hard diffusion coating, applying thin films and surface heat treatments have all been used to enhance the surface properties of steel for more than 100 years. In addition to the common techniques to refine the surface crystalline structure, creating a surface nano crystalline layer is a fairly young process in this area. It has been shown (Masahide, 2003) that Surface Severe Plastic Deformation (SSPD) techniques such as Brushing and Sand Blasting may generate enough intensive strain on the surface layer to create a nano structure of several microns in thicknesses. This hard layer on the steel parts increases the wear resistance and hardness and, as explained previously, may increase the life time considerably.



Moreover, it should be taken into consideration that creating a nano structure on the whole body of a steel part is not always possible or economically favourable.

Increasing the grain boundary density under the SSPD process, increases the diffusion rate in the surface layer. Accordingly, applying SSPD methods prior to post surface treatment techniques such as nitriding, improves the rate and effectiveness of the diffusion processes. For this purpose the thermal stability of the nano structured layers needs to be studied and considered to define the maximum temperature of post heat treatment processes or acceptable working conditions.

### **Problem Statement**

Nano crystallization processes have given us an opportunity to design and fabricate metallic materials with improved physical, mechanical and chemical properties compared with traditional material having micron size crystalline structure. Although various studies have been conducted in this area over past few decades, still it is important to improve our knowledge about nano materials properties and nano crystallization processes to be able to well commercialize these techniques. There are limited studies and reports regarding drilling as a new introduced surface severe plastic deformation nano-crystallization technique (SSPD). Drilling is a simple and low cost technique with easy controllable process parameters which has the capacity of both laboratory and industrial scale applications. Therefore, it could be a valuable research to study drilling technique controlling parameters especially on standard commercial materials such as CK60 steel. Having clear information regarding thermal resistance and mechanical behaviour of materials in elevated temperatures is essential in design process of many industrial steel parts and components.

Considering the available reports and published data in this area, it seems that the study of nano-crystallized steels' thermal behaviour is a new subject which demands more experiments.

## **Objectives**

Bearing in mind the advantages of creating a nano crystalline structure and the importance of the surface treatment of steel, the objectives of this research are:

- x To create a nano-crystalline structure on commercial medium carbon steel (CK60) by using the drilling method as a simple and low cost SSPD technique and to study the controlling parameters.
- x To examine the morphology, microstructure and mechanical properties of the nano crystalline layer.
- x To analyse the thermal stability of a nano crystalline structure.

## **Thesis Outline**

Due to the importance of understanding the background of the thesis and the SPD process, in Chapter 2 some of the SPD and SSPD processes and their controlling parameters are reviewed. A review of previous research studies is undertaken by focusing on mechanical work to carry out grain refinement on metallic materials and also to present a general introduction to hardening, thermal stability and heat treatment of steel.

Chapter 3 highlights the methodology of the study, including materials, sample preparation and the characterization methods applied. Chapter 4 deals with the

results and discussion concerning the characterization of nano crystallized samples and the thermal stability of the nano structures created. Chapter 5 summarizes the results and gives some suggestions for future work.

### **Scope of Work**

The scope of work in this thesis could be summarized in the following main activities:

- x Study drilling SSPD technique as a new introduced nano crystallization method on commercial CK60 Steel
- x Selecting proper drilling bit to archive desired cutting speed
- x Pre-heat treatment influence on nano crystallization process
- x Study the relationship between cutting rate and NC layer thickness
- x Measuring minimum required strain to form nano structure
- x Study the microstructure and mechanical properties of formed NC layer
- x Study the thermal stability of NC layer at temperature range of 400 to 650 °C

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