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**Effect of acid treatment for reducing pores clogging in
polyvinyl alcohol grinding stone**

By

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LIST OF ABBREVIATIONS

HDD	Hard Disk Drive
PVA	Polyvinyl Alcohol
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
NiP	Nickel Platinum
Co	Cobalt
Cr	Chromium
SiC	Silicon Carbide
Al ₂ O ₃	Aluminum Oxide
CBN	Cubic Boron Nitride
MMCs	Metal Matrix Composites
XRD	X-ray Diffraction
EDTA	Ethylenediamine tetra-acetic acid
CO ₂	Carbon Dioxide
EDX	Energy Dispersive X-ray
SEM	Scanning Electron Microscopy
ICDD	The International Centre of Diffraction Data

LIST OF SYMBOLS

2θ Diffraction angle

μ Micro

$^\circ$ Degree

Δ Changes

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LIST OF EQUATIONS

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2.1	$\text{Al}^{3+} + \text{C}_6\text{H}_8\text{O}_7 \rightarrow \text{Al}(\text{C}_6\text{H}_5\text{O}_7) + 3\text{H}^+$	29
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Kesan Rawatan Asid terhadap Pengurangan Pori yang Tersumbat dalam Batu Kisar Polivinil Alkohol

ABSTRAK

Batu kisar yang digunakan untuk proses pengisaran cakera aluminum telah tersumbat oleh timbunan serpihan setelah digunakan selama beberapa kali. Pori yang tersumbat ini telah mengubah struktur permukaan batu kisar sehingga mengakibatkan calar pada cakera aluminum semasa proses pengisaran. Tujuan utama tesis ini adalah untuk mengkaji sifat-sifat batu kisar sebelum (kawalan) dan selepas (sampel) digunakan dalam proses pengisaran serta mengkaji kesan rawatan asid terhadap kedua-dua jenis batu sekaligus mengurangkan pori yang tersumbat dalam batu kisar. Analisis mikrograf dan unsur bahan menunjukkan pori yang tersumbat mengandungi serpihan dari proses pengisaran cakera aluminum. Taburan pori pada batu kisar yang belum digunakan adalah tidak sekata menyebabkan ketumpatan dan kekerasan batu kisar adalah berbeza di setiap kawasan. Serpihan yang lebih besar dari saiz pori tidak boleh melepasi pori yang bersaiz kecil lalu menyebabkan timbunan serpihan yang menjadikan pori tersumbat. Batu kisar telah dirawat dengan tiga jenis asid yang berbeza iaitu asid sitrik, asid fosforik dan asid oksalik pada setiap kepekatan yang berbeza iaitu 0.5, 1.0 dan 1.5 M. Masa rawatan untuk setiap asid adalah pada 1, 30 dan 60 minit. Pori yang muncul setelah rawatan asid telah dikira disertakan dengan struktur mikrograf menunjukkan bahawa asid-asid ini boleh menutup ruang pori dalam batu kisar kawalan tetapi mampu untuk mengurangkan pori yang tersumbat pada batu kisar sampel. Pada kepekatan 1.0 M, asid oksalik tertinggi dalam penyingkiran serpihan yang tersumbat dalam batu kisar sampel. Ini adalah kerana asid oksalik mengekstrak silikon karbida yang terdapat dalam batu kisar yang digunakan untuk mengisar cakera aluminum. Pada kepekatan yang sama, asid fosforik cenderung untuk melabur di dalam pori batu kisar. Kesimpulannya, 1.0 M asid sitrik dan masa rawatan selama 1 minit adalah parameter yang optimum untuk digunakan sebagai cecair pembersih batu kisar yang tersumbat. Selain tidak memberi kesan pada cakera aluminum, sitrik asid adalah ejen pengkelatan yang terbaik untuk mencairkan sisa aluminum dalam batu kisar dan tidak mengkelatkan silikon karbida.

Effect of Acid Treatment for Reducing Pores Clogging in Polyvinyl Alcohol Grinding Stone

ABSTRACT

Grinding stone after aluminum disk grinding was clogged by debris loading after used for a certain period of time. The clogging pores modified the surface structure of grinding stone thus resulting in scratching of aluminum disk during grinding process. The aim of this thesis is to determine the properties of raw (control) and clogged (sample) grinding stone thus reduced the clogging problem in grinding stone using acid treatment. The grinding stone were characterized to understand the factor of pore clogging. Morphology analysis by scanning electron microscope (SEM) and elemental analysis using X-ray fluorescence (XRF) spectrometer found that, the clogging area were consist of debris from aluminum disk grinding. Pore size and distribution in unused grinding stone from micrograph structure were varied and inhomogeneous. Since size of debris cannot be controlled, there were some of debris size that larger than the pore size. Even though pore were connected each other as a tunnel for debris removal, the bigger debris size cannot flow through small hollow of pore which cause the loading of debris resulted in pore clogging. The pore size was not uniform, density and hardness (Shore A Durometer) of control grinding stone also were varied. The grinding stone were treated with three different types of acid (citric, phosphoric, and oxalic acid) at concentration of 0.5, 1.0, and 1.5 M with treatment time of 1, 30, and 60 minutes. Porosity results and micrograph structure of the grinding stone indicated that the acids can caused clogging in control grinding stone but had the ability to produce pores for sample grinding stone. At concentration of 1.0 M, oxalic acid shows the highest clogging removal compared to citric and phosphoric acid. However, elemental analysis by energy dispersive x-ray (EDX) spectroscopy shows that oxalic acid tend to remove silicon carbide (SiC) abrasive of grinding stone and phosphoric acid has the tendency to deposit in grinding stone. As conclusion, 1.0 M citric acid and stirring time of 1 minute was the optimum parameter for dissolving the pore clogging in sample grinding stone. Besides not affecting aluminum substrate, citric acid also the best chelating agent to dissolute aluminum debris in grinding stone while does not chelating with silicon carbide.

CHAPTER 1

INTRODUCTION

1.1 Aluminum Grinding

Hard disk drive (HDD) become the most preferable for online data storage as the price offered was reasonable, easy to access for data transfer and storage. This new era of technology is demanding for inexpensive HDD with greater capacity for storage and quick handling of data transfer. The device such as MP3 player, video cameras, video recorder, games consoles and many other devices use HDD to store the information. Even though most of the MP3 nowadays use the flash memory device, video camcorder and recorder still used HDD due to the requirement of larger storage capacity (Piramanayagam & Srinivasan, 2009). Thus, the manufacturing of main component in HDD which is magnetic disk substrate become the most crucial part in industry as to achieve the technology demand. The material for manufacturing of magnetic disk substrate must be very slim, light and low inertia as the disk can rotate rapidly without consuming more power as to avoid too much heat generation (Wood, 2009; Yeh et al., 2014).

As the demanding of hard disk drive is increasing, the requirement for hard disk drive to operate efficiently becomes higher. Thus, the roughness and defects on the surface of magnetic disk substrate must be minimized as to avoid head crashes, damage to read or write

head which can damage the data stored in the disk's magnetic coating (Lei et al., 2010; 2014). The magnetic disk substrate that made up from a high purity aluminum alloy is more beneficial compared to other alloy because of its light weight properties. Aluminum alloy in a form of roll coil is punched into the small scale to prepare the blank. The blank will be ground in order to obtain the flawless surface. After polishing, the substrate will be coated with magnetic film by magnetic sputtering (Tada et al., 2000; Wood, 2009).

1.2 Grinding

Abrasive are important in grinding process as to facilitate the removal of unwanted surface in substrate. The usage of abrasive in grinding process can be divided into two ways. First, the abrasive is attached into the polishing or grinding pad with the combination of other material such as resin as the binder. Second, the slurry or coolant during grinding process was manufactured with the abrasive particle (Lei et al., 2012).

Since aluminum alloy is very soft, conventional lapping machine for grinding which uses cast iron plate and free abrasive will cause the serious quality problem of the magnetic substrate (Tomita & Eda, 1996). The surface finish and surface integrity are important for surface-sensitive parts subjected to fatigue or creep. A proper grinding process is needed to obtain a good surface finish and damage-free surface for better application of the materials (Zhong & Hung, 2002). Thus, to perform a new process by using a double-sided lapping machine with grinding stone, a new bonding resin which fixes free abrasive grains in the matrix of the grinding stone is developed (Tomita & Eda, 1996). The grinding process is carried out by a rotating action of the grinding stones and by a planetary motion of the work

piece in the presence of grinding fluid as shown in Figure 1.1 (Tomita & Eda, 1997). The surface grinding of metal plates, glass sheets, synthetic resin plates and such other materials were often performed by a lapping machine. The machine was provided with a plate like lapping platen or a lapping platen having bonded with artificial suede leather. A material to be grind is placed against the lapping platen, grinding slurry containing abrasive grains is continuously supplied, and the material is sliding on the lapping pattern (Sato et al., 1991).

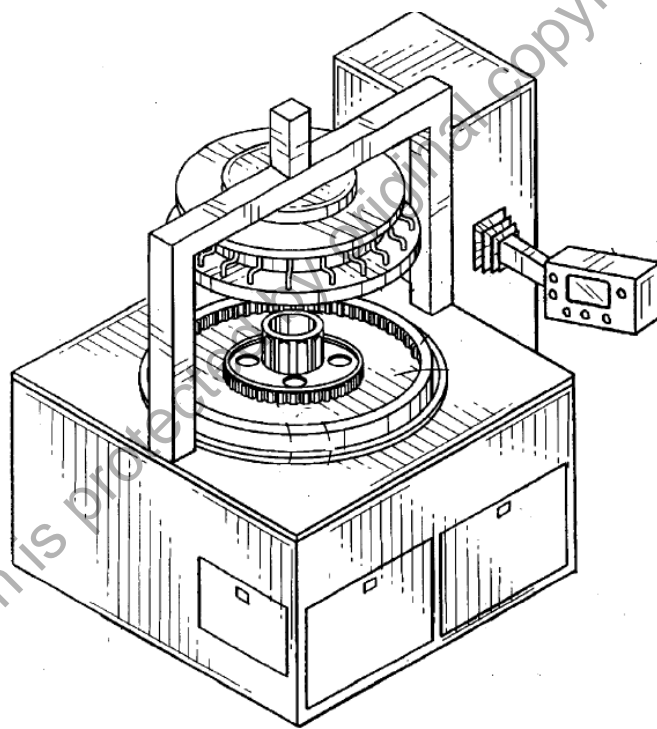


Figure 1.1: A Perspective view of grinding machine (Lichner, 2000).

Grinding stone made up from polyvinyl alcohol (PVA) resin is suitable for grinding of soft materials such as aluminum, soft steel and stainless steel. The mixture of PVA resin and abrasive without other thermosetting resin in manufacturing of grinding stone was a failure. The mechanical strength of grinding stone was low because the adhesion between

abrasive grains and binder is poor. Other than that, the pore formation also quite large and does not have a uniform distribution (Kagawa, 1975). Then, a new mixture of PVA resin, melamine and phenol resin (thermosetting resin), fine grain of silicon carbide (abrasive), formaldehyde (hardener), sulphuric acid (catalyst) and pore forming chemical were introduced in grinding stone manufacturing. This new mixture of grinding stone were adjusted its mixing ratio to obtain the best quality of grinding stone (Tomita & Eda, 1996).

Grinding fluid functions as lubricating, cleaning, penetration, cooling and anti-oxidizing actions. Besides that, oxidization of fine debris aluminum can be prevented by using grinding fluid. Thus, loading of debris on the stone surface is also prevented. The grinding of the ground surface is mainly influenced by grades and features of the grinding stone. However, it is also greatly influenced by the features of grinding fluid. In particular, the grinding efficiency, consistency, productivity and continuity of grinding ability are remarkably influenced by grinding fluid (Tomita & Eda, 1997). The grinding fluid can be tailored depending on the choice of oxidizing agent and the abrasive in order to provide the effective of polishing rate as well as minimize the surface defect such as scratches, corrosion and erosion (Kaufman & Wang, 1998). Some of oxidizer which used in the polishing slurry were hydrogen peroxide, nitric acid, acetic acid, phosphoric acid and citric acid (Lin et al., 2005; Faller & Cadien, 1997; Tsai & Kuo, 2001).

1.3 Problem Statement

Grinding stone performance during grinding is not long lasting. The grinding fluid becomes more viscous and concentrated. The concentrated grinding fluid containing debris enters into fine structure of grinding stone. The porosity of the grinding stone starts to clog with the debris and residues from the aluminum magnetic substrate after usage for a certain period of time. The clogged pore in the grinding stone will hamper the removal of debris and flow of grinding fluid. The debris then will stack in the pores thus adding another frictional force beside the ongoing frictional force from the abrasive in the grinding stone which may increase the roughness to the grinding substrate (Tomita & Eda, 1996). The harder part of the stone will caused the scratches on the aluminum magnetic substrate during the grinding process. The scratches also agitated the properties of the aluminum disk, thus affect its performance. The cooling effect function from the grinding fluid may also be disrupted. The problem occur due to the pores clogging can decreases the grinding efficiency and also decreases the tool life of the grinding stone (Hou et al, 2012). This is because the entire surface of grinding stone must be uniform to obtain a good quality grinding substrate. Even though the grinding stone surface was worn away, it is desirable to maintain the properties of grinding stone equivalent to the new one in term of flat surface and hardness (Fruitman et al., 1998).

Dressing the worn out grinding stone surface can increased the grinding efficiency. However, dressing also can resulted in excessing grinding stone material loss by fractured the bond and breakage of abrasive grain (Jackson et al., 2007). Optimization of process parameter, grinding fluid supply, tool design and tool conditioning can reduced the grinding

stone clogging. High pressure grinding fluid supply can be used to prevent clogging of grinding stone. However, for industrial application low pressure and flow rates would be favorable (Heinzel & Antsupov, 2012). Addition of acid in grinding fluid was invented to increase grinding efficiency by cleaning the grinding stone (Hamill & Mallon, 2008). Acid such as citric and oxalic acids has an ability to dissolve metal and widely used in cleaning process because it has capability to chelate metal ion. (Ataman, 2005). Since citric, oxalic and phosphoric acid have the properties that can extract metals, various parameter of the acids were investigated in this project to remove the clogging pores in grinding stone. At the end of finding, the acid treatment was expected to remove the debris clogging in the shortest treatment time without damaged the grinding structure will be choose as the optimum parameter for debris removal in clogging PVA grinding stone.

1.4 Research Objectives

The objectives of this research proposal are:

- 1) To investigate the pores properties of control PVA grinding stone and sample PVA grinding stone after grinding operations.
- 2) To investigate the effect of different acids with different molarities and stirring times on the pores debris removal from control and sample PVA grinding stone
- 3) To find the optimum parameter for debris removal using acid solution in the shortest treatment time without damaged the structure of PVA grinding stone.

1.5 Scope of Study

The study of grinding process parameter and material involved are needed as to determine the cause of grinding stone clogging and scratches on aluminum disk. In order to improve the process parameter, it is believe to do the research works on the relation of grinding material, aluminum and grinding fluid. The treatment process of grinding stone and aluminum disk in the acid solution were carried out by magnetic stirrer hotplate at a varying parameter such as types of acids, acid concentration and treatment time. The acid chosen were citric acid, phosphoric acid and oxalic acid. The concentration of the acid were varied from 0.5, 1.0, and 1.5 M. The treatment time are from 1 min, 60 min and 1 hour. The density before and after treatment will be measured to find the changes in porosity value. The sample before and after treated in the acid will be characterized by various analysis such as X-ray diffraction, morphology analysis, porosity changes, hardness and ICP-AES.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Aluminum is the second most abundant metal on earth, and it has widely used in most industrial application (Davis, 1999). Aluminum becomes an astonishing economic and industrial importance due to its low cost, high thermal and electrical conductivity, low density, high specific strength, ease of casting and reasonable corrosion resistance (El-Etre, 2001; Altintas et. al., 2015). Aluminum has a good corrosion resistance because of thin oxide film formation when it is exposed to the atmosphere. When aluminum is exposed to the acidic or alkaline condition, it will destroy the coating layer and caused the corrosion. Phosphoric acid is the most preferred in electropolishing aluminum and metal cleaning compared to other acid such as sulphuric acid. This is because aluminum oxide particles are soluble in phosphoric acid solution (Rao & Prabhu, 2013). Besides, aluminum also has an attractive appearance in its natural finish, recyclable and non toxic which can be used as food and beverage container (Davis, 1999).

Since aluminium has many benefits, it becomes important in many industrial developments. It can be processed into automotive material, lightweight conductive metal, household item and substrate in technology devices (Davis, 1999; Gopalan et. al., 1998) Aluminum alloy is widely used in automotive and aerospace industries because it has good

mechanical properties, low density and good in corrosion resistivity. The metals also are suitable for production of hydrogen can be utilized into energy materials. Aluminum becomes a very attractive material for energy storage and conversion due to high capacity per unit weight (2980 Ah/kg) and per unit volume (804 Ah/l) (Mokaddem et al, 2010).

2.2 Aluminum Magnetic Disk Grinding

Grinding is the process of cutting material by using abrasive to remove the unwanted surface of the grinding substrate. The unwanted material will be removed by the lapping surface of grinding wheel and the substrate of grinding. The suitable grinding tools must be consider to ensure that the machining cost is efficient and the surface quality of the substrate must be better (Krajnik & Kopac, 2006).

Grinding is carried out by a rotating action of the grinding stones and by a planetary motion of the workpiece in the presence of grinding fluid. The surface grinding of metal plates, glass sheets, synthetic resin plates and such other materials were often performed by a lapping machine. The machine was provided with a plate like lapping platen or a lapping platen having bonded artificial suede leather. A material to be grind is pressed against the lapping platen, grinding slurry containing abrasive grains is continuously supplied, and the material is sliding moved on the lapping pattern (Sato, 1991; Lichner, 2000).

A magnetic disk used in memory hard disk were made up from aluminum alloy, plating with NiP and other magnetic coating layer was grinding and polishing to obtain a smooth surface. There were many layer of magnetic layer to overcoat the disk. Examples of