NOVEL FORMULATION OF WASTE RUBBER BINDER IN PRODUCING NET SHAPED AND PRECISE M2 HSS COMPONENTS

RESEARCH MANAGEMENT INSTITUTE (RMI)
UNIVERSITI TEKNOLOGI MARA
40450 SHAH ALAM, SELANGOR
MALAYSIA

BY :

NOR 'AINI BT WAHAB
MOHD AFIAN OMAR
ISMAIL NASIRUDDIN AHMAD
NOR AMALINA NORDIN
AMALINA AMIR

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Contents

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PROF. DR. ABU BAKAR B. ABDUL MAJEED
PENOLONG NAIB CANSELOR (PENYELIDIKAN)
INSTITUT PENGURUSAN PENYELIDIKAN (RMI)
UNIVERSITI TEKNOLOGI MARA, UiTM
40450 SHAH ALAM

Yg. Bhg. Prof.

LAPORAN AKHIR PENYELIDIKAN "FUNDAMENTAL RESEARCH GRANT (FRGS):
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Dengan hormatnya perkara di atas adalah dirujuk.

Bersama-sama ini disertakan 1 (satu) CD yang mengandungi laporan akhir penyelidikan bertajuk **"** **Novel Formulation of Waste Rubber Binder in Producing Net Shaped And Precise M2 HSS Components "** yang telah tamat dijalankan oleh kumpulan penyelidik dari FKM untuk simpanan dan tindakan pihak Prof seterusnya.

Sekian. Terima kasih.

Yang benar

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2. Prof. Madya Dr Ismail Nasiruddin Ahmad, Ahli Projek, Fakulti Kejuruteraan Mekanikal, **UiTM Shah Alam**
3. Pn Nor Amalina Nordin, Ahli Projek, Fakulti Kejuruteraan Mekanikal, **UiTM Shah Alam**
4. Pn Amalina Amir, Ahli Projek, Fakulti Kejuruteraan Mekanikal, **UiTM Shah Alam**
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(if any)

Original Title as Proposed:

Novel Formulation of Waste Rubber Binder in Producing Net Shaped And Precise M2 HSS Components

Improved/Enhanced Title: None

Original Objectives as Proposed:

1) To assess the possibility of utilising abundant waste rubber as one of the binder components in Metal Injection Molding of M2 HSS.

2) To analyze the molding behaviours and properties of the moulded parts

3) To evaluate the physical and mechanical properties of the sintered parts

Improved/Enhanced Objectives: None

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5. Report

5.1 Proposed Executive Summary

Metal Injection Moulding is an attractive process especially for small, precision parts combined with complicated shapes [1-2]. It combines the advantages of net-shaped components of powder metallurgy (P/M) and easy shaping of plastic injection moulding. The process starts with mixing of metal powder and binder to form a homogenous feedstock, followed by injection moulding in order to shape the feedstock. Binder is then removed completely by a debinding process and finally sintered to consolidate the powder.

Many studies have been carried out in developing a novel binder system in MIM with the main aim of shortening the overall debinding time and at the same time maintain shape integrity during subsequent processing [7]. In previous study conducted by A. Amalina et. al., 17-4 PH(2009) stainless steel powder has been successfully moulded with binder consisting of polyethylene (PE), paraffin wax PW), palm stearin (PS), thermoplastic natural rubber (TPNR) and stearic acid (SA) to form a homogenous feedstock.

In this proposed research work, the main aim is to formulate a novel binder system which may improve the functional properties and performance of M2 High Speed Steel (M2 HSS) in demanding applications and thus increase the competitiveness of M2 HSS versus cemented carbides primarily in machining operations. As Malaysia produces hundred tonnes of natural rubber every year which has been utilised in production of rubber based products it is hoped that waste rubber which is currently underutilised can be used as one of the binder components in metal injection moulding of M2 HSS.

It is also hoped that the abundant supply of waste rubber could be utilised to produce a novel binder system which is desirable for both economic and environmental reasons. The novel binder system could be patented and can be used in local manufacturing industries producing small, high precision and complicated shapes of metallic parts.

5.2 Enhanced Executive Summary

(Abstract of the research) – 1 page only

Reseach has been performed to develop a new binder that will allow powder metal injection moulding (MIM) of Molydenum High Speed Steel (M2 HSS). The development of the new binder parallels that of other binders utilised in MIM of other metal powder such as stainless steel, cemented carbides, tungsten heavy alloys, and cobalt based alloys. The binder/metal powder ratio, the mixing procedure, the injection moulding process, the debinding process and sintering process have been explored experimentally to optimise the MIM process using M2 HSS as the metal powder and waste rubber (WR) as one of the binder constituent

Injection moulding is a known and viable net-shape process for manufacturing small, complex shaped components, especially for high performance applications. Feedstock preparation for MIM is a vital step as shortcomings such as inhomogeneity of feedstock, metal particles segregation and metal powder-binder seperation cannot be corrected by subsequent processing adjusment. One key component in MIM is the selection of binder and the formulation as the binder promotes fluidity and rigidity of the feedstock particularly during mixing, injection moulding and debinding. The characterictics of the binder greatly affected MIM parameters such as particle packing, agglomeration, mixing, rheology, moulding, debinding, dimensional accuracy, defects and properties of the sintered MIM part

Two binder systems have been successfully developed for MIM of M2 HSS components. The binder systems comprise of waste rubber binder combined with major fraction of conventional binder, paraffin wax and local binder based on palm oil derivative palm stearin. Paraffin wax (PW) and palm stearin (PS) can be removed rapidly by heptane leaching without deterioration of the green injection moulded parts. Polyproplene (PE) can be removed by prolysis during ramping up to the sintering temperature.

Studies have been performed to the feedstock compositions and to the process parameters in order to obtain high strength and dense components. Feedstocks having a powder loading of the 22um M2 HSS mixture up to 65 vol% can be injection moulded successfully. The developed binder systems provide flow ability of the M2 HSS particles and shape retention of the moulded parts. The brown parts were then sintered in vacuum atmosphere within a temperature range of 1200°C to 1260°C. Optimum sintered properties were obtained from the conventional binder, paraffin wax, whilst the sintered properties obtained from local binders palm stearin are comparable and good. The paraffin wax binder sintered at 1250°C possessed maximum density of was 8.095g/cm^3 whilst palm stearin binder achieved

optimum density of 8.111g/cm^3 when sintered at 1240°C. The results suggested that the sintered density obtained from both binder systems have exceeded the theoretical density. The results also showed that optimum strength of paraffin wax and palm stearin binder were 2351Mpa and 2210MPa respectively, both recorded at sintering temperature of 1230°C. The strength of sintered parts for both binder systems decreases with increasing sintering temperatures which could be due to embrittlement of the part as a result of grain growth effect.

5.3 Introduction

Product cost effectiveness has been the predominant reason for choosing Powder Metallurgy (PM) and is the main driver of the structural (or mechanical) parts sector. Due to its lower energy consumption, higher material utilisation and reduced numbers of process steps compared with other manufacturing routes, PM has emerged as cost effective manufacturing technique for relatively small, complex and high performance components. One of such approaches in meeting these needs is the development of metal injection moulding (MIM).

Metal Injection Moulding has evolved to become a versatile mass production method for a wide range of net- and near-net shape complex metal components for the medical, industrial and consumer industries[1]. It combines the versatility and high productivity of the plastic injection moulding with the powder metallurgy technique of sintering. The process which consisting of four sequential technological processes; mixing, injection moulding, debinding and sintering can be a cost competitive manufacturing route, especially when compared with other conventional routes; powder metallurgy press and sinter, invesment casting or machining (Figure 1).

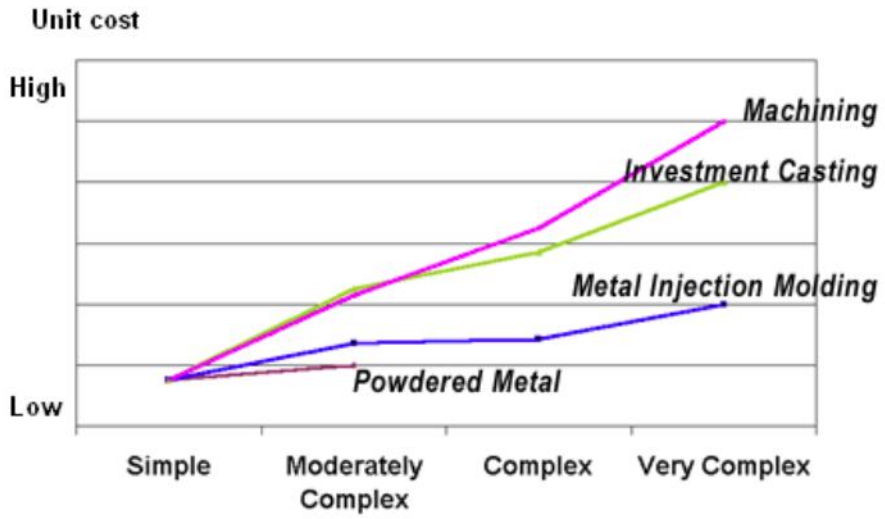


Figure 1.1: Comparison of manufacturing methods as influenced by specimen cost and complexity (SSI Technologies, Inc., 2008)

Since the commercial beginning of the MIM process in the mid 1970's, the technology has evolved to become a versatile mass production technique for a wide range of complex-shaped metal components (SSI Technologies, 2008). The MIM components which are typically 95% to 98% dense, possessed greater strength, better corrosion resistance and enhanced magnetic properties compared with components manufactured by conventional powder metallurgy processes.

MIM process starts by combining a small quantity of metal powder with binder to form a feedstock that can be moulded into complex shapes. The binder, which acts as temporary vehicle to ease flowing is then removed from the green specimen through various debinding processes. The brown specimen produced retains its shape as a result of greater inter-particle friction. At this stage, the specimen is very brittle and

needs to be handled with great care particularly when uploading in the sintering furnace. Sintering is carried out to achieve its final sintered density and the desired mechanical and dimensional properties.

One of the main issues in MIM is the selection of a suitable binder system. The binder system designed should be able to provide flow ability, good packing of the metal particles and good mechanical strength. Many studies have been carried out in developing a novel binder system in MIM with the main aims of shortening the overall debinding time and at the same time remain shape integrity during subsequent processing [6-18].

Omar et al. [16-18] reported that homogenous feedstock prepared by using palm stearin (PS) and polyethylene (PE) can be injection moulded and sintered successfully. The sintered parts produced do not contain any defects and has density comparable with the theoretical density. The bio-polymer binder used which is locally available is not only economical but also environmental friendly. In addition, it has been proven that this binder system can shorten the overall debinding process by performing solvent extraction technique.

In a recent study conducted by A. Amalina et. al.[24-25], 17-4 PH stainless steel powder has been successfully moulded with binder consisting of polyethylene (PE), paraffin wax PW), palm stearin (PS), thermoplastic natural rubber (TPNR) and stearic acid (SA) to form a homogenous feedstock. It has been reported that this versatile, organic locally binder system is cheaper, safe in practice, environmental friendly and present fewer health hazards to workers and environment during processing of metal components.

Z.Y.Liu et. al. has reported that the production of high speed steel (HSS) parts by MIM offer greater enhancement in wear resistance, hardness, heat resistance, toughness, and dimensional stability compared with conventional cast or wrought parts. This is explained by the fact that MIM parts have more refined and homogenous microstructure.

As Malaysia produces hundred tonnes of natural rubber every year which has been utilised in production of rubber based products it is hoped that waste rubber which is currently underutilised can be used as one of the binder components in metal injection moulding of M2 HSS. It is also hoped that the abundant supply of waste rubber could be utilised to produce a novel binder system which is desirable for both economic and environmental reasons.

5.4 Brief Literature Review

Metal injection moulding (MIM) is an economically attractive method that combine the productivity of injection moulding with the versatility of sintering of metal particulates in producing large amount of small, complex and high performance metallic components. The process consists of four main stages; preparation of feedstock by mixing powdered metal with binder, injection moulding of the feedstock, removal of the binder from the feedstock (solvent and thermal debinding) and sintering of the metal powder.

The MIM process does not usually require secondary working operations owing to its ability to provides net shape components. The key point in MIM turned out to be how to ensure the metal flow into the mold cavity and consequently retain the shape of the injection moulded part until the beginning of the sintering stage. This problem is commonly solved by dispersing the powdered metal into a binder to form a paste that flows at high temperature and becomes solid at room temperature. As a results, the injection moulded part retains its shape following the injection moulding and may be handled and processed safely.

Figure 2 illustrates schematically MIM process that includes four major processing steps: feedstock formulation, injection molding, debinding, and sintering.[23]

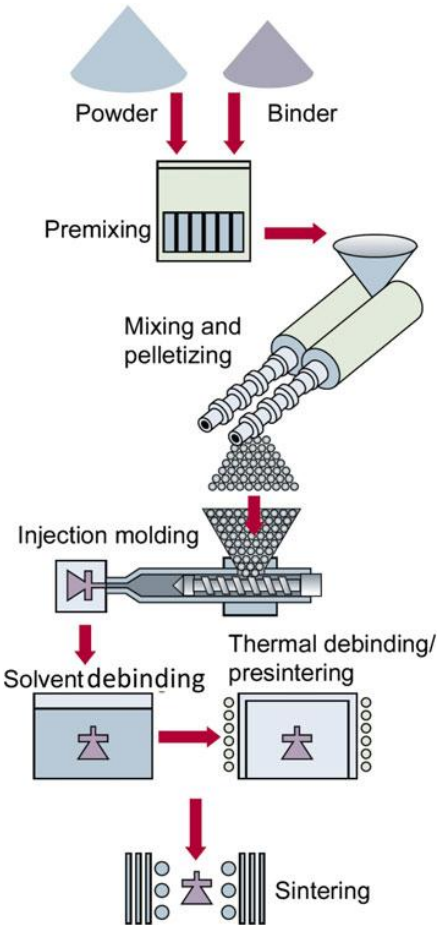


Fig. 2—Schematic of generic metal injection molding processes: feedstock formulation, injection molding, debinding, and sintering: [23] .

The MIM process starts with the preparation of a homogeneous powder feedstock by mixing metal powders with a suitable binder system. The blended powder mix is worked into the plastified binder at elevated temperature in a kneader or shear roll extruder. The intermediate product is the so-called feedstock. It is usually granulated with granule sizes of several millimetres, as is common in the plastics injection moulding industry.

The formulation of the feedstock (metal-binder mixture) is one of the most crucial aspects due to its effect on every step of the MIM process. Low amount of binder used in formulation of the feedstock resulted in high viscosity feedstock which making the moulding process difficult. On the other hand, large amount of binder provides low strength and may produce heterogeneous green parts.

Following the injection moulding stage, the wax/polymer binders are eliminated from the green part by either, solvent extraction followed by thermal treatment, or by thermal debinding alone. Solvent debinding takes advantage of the high solubility of low molecular weight constituents (major binder) in organic solvents. It has been well established however, that leaching or extraction of one or various binder components creates porosity in the green part. The open porosity created during leaching allows gasses resulting from decomposition of the minor binder to diffuse to the surface easily [2]. Hence, the thermal removal of insoluble binder components could be accomplished in a much shorter period without endangering the integrity of the green part as generation of internal stresses is reduced significantly [10].

At the end of the debinding process there is often still some binder present in the parts holding the metal powder particles together. The pore network present in the debound part allows the residual binder to evaporate quickly in the initial phase of sintering at the same time as sintering necks start to grow between the metallic particles.

The sintering process leads to the elimination of most of the pore volume formerly occupied by the binder. Consequently, metal injection moulded parts exhibit a substantial shrinkage during sintering. The linear shrinkage is usually as high as 15 to 20%. During this process, the individual particles metallurgically bond by solid state diffusion resulting in enhancement of mechanical, physical and dimensional properties of the part. The sintering process shrinks the part, providing a net shape that can be used as-is or further worked to add additional features or improve tolerances.

The homogeneity of a powder feedstock is crucial in MIM processing to avoid any inhomogeneity, including bubbles, left during the mixing step will not be carried over to the subsequent processing steps. An inhomogeneous feedstock usually leads to a poor flow behavior during injection molding stage, which consequently resulted in low densification and poor dimensional stability in the final injection molded products. The selection of binders in MIM is usually based on considerations of producing homogenous feedstock which is free of binder separation or particle segregation in order to offer high quality injection molded product. Typically,the binder present in the feedstock acts as a temporary vehicle for shaping the feedstock into the required geometry and holding the particles in that shape until the start of sintering stage.

Binder formulation plays a vital role in MIM. The binders used in MIM are usually a multi-component mixture in order to ensure a defect-free MIM parts can be produced [11]. According to MIM literature [1-11] the binders may be broadly categorised into two groups; those based on wax/polymer or, on polymer/polymer compounds. Common binders in MIM are wax-based binder systems and the common backbone

polymers are polyethelene and polypropylene. The function of polymer is to provide the shape retention during the following steps in the global process, while wax reduces the viscosity of the feedstock and allowing good flowability during the injection moulding stage. In addition, the polymeric component which is also known as backbone component provides sufficient brown strength to the injection molded component after the major component is removed during the debinding stage. In most cases, the binder system also contains a third component, such as a surfactant, which serves to enhance compatibility between the metallic powders and the polymer. A common surfactant as stearic acid increases the wettability and miscibility, thus reducing separation and segregation of metal particles [3]. Despite the great progress that has been achieved in binder formulation, it is crucial to ensure that all the binder components are compatible with each other.

Hard metal such as Molydenum High Speed Steel (M2 HSS) have many attractive properties such as high wear resistance, high toughness, good red hardness and retains its cutting edge longer than other general purpose high speed steels. Despite the superior characteristics compared with the older high-carbon steel tools used extensively through the 1940s, M2 HSS is very difficult to produce by melting and casting and are often very brittle in the cast state.

Such metals are The production M2 HSS by MIM is considered to be better than other manufacturing technique due to the inherent capacity of this technique to produce near net shape components, avoiding costly machining and obtaining finer and more controlled microstructure which in turn provides superior toughness and cutting tool performance compared to wrought products. The principal difficulty presented by this steel is the complex densification process by SLPS (supersolidus liquid phase sintering) [3-4].

During the sintering process the high speed steel undergoes an unusual melting process; the liquid forms on the particle contact and grain boundaries and particle rearrangement causes rapid densification. A critical fractional coverage of the liquid on the grain boundaries muat be achieved to enables this process to takes place. The sintering temperature and the carbon content are the most vital variables in this process sice these dictate the volume fraction of the liquid phase [ ]. Accurate control of the temperature and composition leads to accurate control of the volume fraction of liquid which in turn leads to full density, avoidance of shape distortion and minimization of microstructure coarsening. The densification process and the microstructure development are the parameters for controlling the sintering window (temperature region in which optimum sintering takes place) because it is very narrow for M2 HSS [3-4,6].

Prior study by Liu et.al. revealed that M2 HSS has been successfully injection moulded by using a multi-components binder consisting of natural wax, fatty acid wax, stearic acid, poly-oxi-alkylen ether and olefin-hydrocarbons. The sintered injection moulded part posssed density approaching the theoretical density when sintered in vacuum environment. Natural polymers which have gained widespread use in different industries has raised interesting issue related to environmental concerns. Conventional wax-based binders have proven their suitability as a binder sytem in production of intricate shape metallic component by using MIM route. However, the injection molded components which ussually suffer long debinding time make them susceptible to defects formation. In addition, extracting the wax from the green components may contribute to toxicologic and environmental concerns. The

focus in recent years thereby has mainly been on development of new environmentally friendly and safer binders.

The search for binder system for M2 High Speed Steel never stopped. Herranz et. al. demonstrated the development of a new feedstock formulation for MIM of M2 High Speed Steel using a wax-High Density Polyethylene (HDPE) binder. The binder is a multicomponent system based on High Density Polyethylene (HDPE) and Paraffin Wax (PW). The injection moulded parts sintered between 1210 and 1280^0C in high vacuum atmosphere demonstrated the 98% of the theoretical density. A homogenous distribution of M_6C and V-rich carbides occured during the sintering stage reinforced the HSS and hence increasing the mechanical properties of the part. In addition, the sintered part also showed good shape retention and minimal dimensional deviation.

Binder compositions and debinding techniques are the main differences between various powder injection moulding processes. Several classes of binder have been used but most have long debinding times which affected the economics of these processes. Utilization of waste rubber as one of the binder component in MIM has received great attention due to the advantage of renewability, thermal stability and high shear viscosity (Tan et.al., 2008). In addition, the newly developed binder system used in MIM processing also exhibit economical and enviromental friendly characteristics especially for the application in automotive, tooling, medical and hardware components.

In the present study, a high density polyethelene (HDPE)-wax based binder containing waste rubber (WR) was formulated and developed. Investigations on the feasibility of the new developed binder were performed by preparing feedstock at solid loading of 65-volume % M2 HSS, kinetic solvent extraction by means of n-heptane followed by sintering at various temperatures in vacuum atmosphere.

5.5 Methodology

This research was conducted at three sequential phases which are characterization of materials, sample preparation and sintered parts characterizations. The details of the research work is summarized in Figure 3.

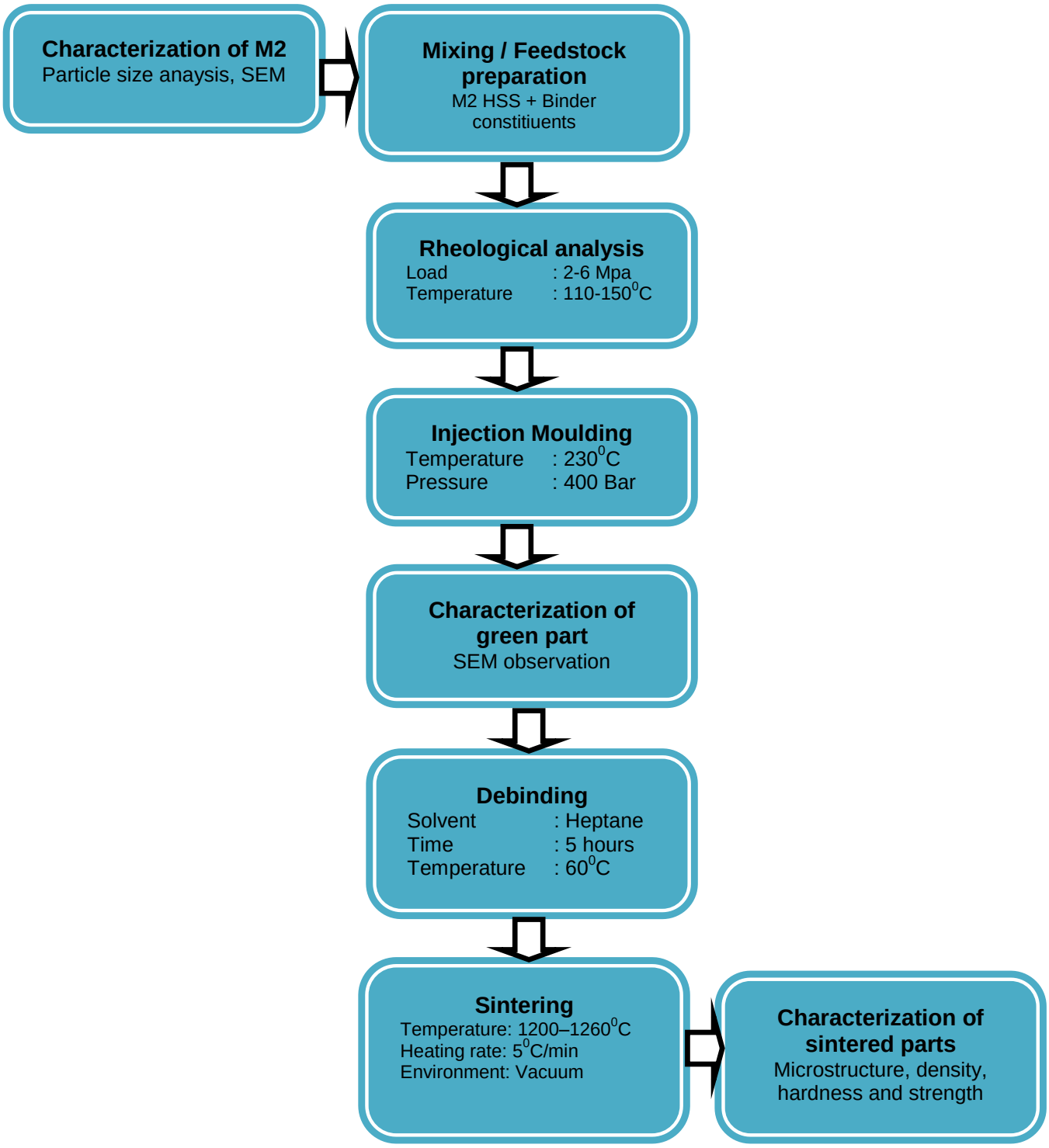


Figure 3 : Flow chart of research work

5.5.1 Materials

In this research work, the main raw material used is Molybdenum High Speed Steel (M2 HSS) which has a mean diameter particle size of 16µm and the binder constituents comprising of Paraffin Wax (PW), High Density Polyethelene (HDPE), Waste Rubber (WR) and Stearic Acid (SA). The feedstock was prepared at a powder loading of 65 vol.% of M2 High Speed Steel powder and 35 vol.% of the binder system based on research work conducted by previous researcher (Omar and Subuki, 2006).

5.5.2 Materials characterization

The gas atomised M2 HSS powders used in the present study was obtained from Sandvik Osprey Powder. The particle size distribution of the of commercial Molybdenum High Speed Steel (M2 HSS) powder was measured using Coulter LS 130 Laser Particles Size Analyzer while the particle morphology was observed using Scanning Electron Microscopy (SEM).

5.5.3 Specimen Preparation

The specimens were prepared through four sequential technological processes consisit of mixing, injection moulding, debinding and sintering; which each significantly affect the characteristics of the final parts. Mixing process of the metal powder and binder system was carried out in a sigma blade mixer for 2 hours at 160°C before it was removed from the bowl, cooled and then granulated into a feedstock. The granulated feedstock was molded into tensile bars by using MCP HEK-GMBH vertical injection moulding machine. In order to obtain defect-free molded parts, a suitable set of molding parameters were established. Injection molding temperature and pressure were 230 °C and 400 bar respectively.

The rheological study of feedstock was conducted using Capillary Rheometer Shimadzu CFT-500D. The rheological characteristics of the fine feedstock was analyzed based on Flow Rate, Shear Rate, Viscosity and Melting Flow Rate. A rheometer capillary die with 1.5 mm diameter (D) of and 10mm length (L) was used in this study. Sample of 10gm was placed in the rheometer barrel and allowed to preheat for 120s at varying temperatures of 110°C, 120°C, 130°C, 140°C and 150°C and under shear stress of 2,3,4,5 and 6 MPa test load before initiating the test.

The green molded parts were subjected to a solvent extraction process where around two third the volume fraction of the binder was removed. Solvent immersion debinding was carried out by immersing the parts in *n*-heptane at 60°C for 5 hours without stirring. The dimension of green body specimens were measured prior to the solvent extraction process. A glass container was used to cover the bath to prevent evaporation of the n-heptane during the extraction process. Upon completion of the solvent extraction process, the brown parts were dried in an oven at a temperature of 40°C for 2 hours to remove the remaining n-heptane. The brown parts were weighted to calculate the amount of binders removed and Scanning Electron Microscopy observation was performed to observe the fracture surface of parts debound for different elapsed times

Following the leaching process, the brown parts were sintered in vacuum environment. Sintering was performed at different temperatures between the ranges of 1200°C to 1260°C in a controlled vacuum atmosphere. In order to prevent

cracking, warping defect and excessive grain growth, the heating rate used was 5°C/min and the specimens were soaked for 2 hours and subsequently furnace cooled.

5.5.4 Density test

ASTM 328 is a density determination standard to measure the density of sintered injection moulded parts by determining the specific gravity using the Archimedes principle. The density measurement was performed by measuring the weight of sintered part in both air and water, without impregnation done to the part, prior to the measurement. The density value was calculated by using the following mathematical equation;

Density, $\rho = [W_{air} / (W_{air} - W_{water})] X \rho_{water}$

Where;
W_{air} = weight of part in air
W_{water} = weight of part in water
ρ_{water} = density of water

5.5.5 Three Point Bending Test

The transverse rupture strength (TRS) of sintered specimens was measured by three point bending test by using Instron Universal Instrument. Transverse rupture strength (TRS) test was performed according to MPIF 41. Ultimate stress and Young's Modulus were measured to evaluate the mechanical properties of the sintered part.

5.5.5 Hardness Test

Hardness measurements were performed by using Rockwell Hardness Test. In this test diamond indenter was used to formed indentation against the surface of the part under established machine setup. The test allows measurement of the depth of the indentation according to the MPIF Standard 51 (under the specified conditions of pleminary and total test forces) to determine the hardness of the sintered part.

5.5.6 Microstructure Observation (SEM)

The as sintered parts were prepared for optical microscopy by standard metallographic techniques and etched in a 5% Nital solution, which preferentially etches the detail microstructure. The cross sectional surface of of parts debound for different elapsed times and and sintered parts were examined by Scanning Electron Microscopy (SEM) by using secondary and backscattered electron mode image detector.

5.6 Results and Discussion

5.6.1 **Materials characterization**

The gas atomised M2 HSS powders with mean particles size of around 16µm used in the present study was obtained from Sandvik Osprey Powder. Table 1 shows the particle size distribution characteristics d10, d50 and d90 of the powder feedstock measured using Coulter LS 130 Laser Particles Size Analyzer.

Table 1: The Cumulative Particle Size Distribution of M2 HSS Powder.

Fraction (%)	< 10	< 50	< 90
Size (µm)	2.14	6.20	16.55

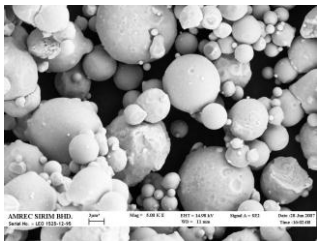


Figure 4. Scanning electron micrograph of commercial M2 HSS powder.

Particle morphology of commercial Molybdenum High Speed Steel (M2 HSS) used in the study is shown in Fig. 2. As depicted in Fig.2, the particles have spherical morphology which leads to high packing density. This characteristic enables production of feedstock with high powder loading, which may minimizes shrinkage of the injection moulded part as a result of debinding and sintering. In addition, sperical M2 HSS powder also enables excellent flow characteristics, resulting in minimum tool wear and consistent mould filling.

5.6.2 **Mixing / Feedstock preparation**

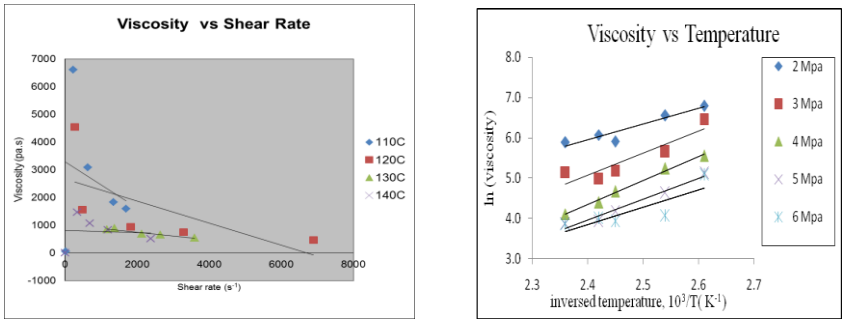
The M2 HSS powders were compounded to feedstock using a proprietary multicomponent binder system as shown in Table 2. The binder components were mixed with the metallic powder at a temperature of 160°C for duration of 2 hours in a Z-blade mixer.

Table 2: Composition of PW/PS/ WR/PE/SA in different feedstock (mass fraction, %).

Feedstock	PW	PS	WR	PE	SA
PW	55	-	14	21	10
PS	-	55	14	21	10

5.6.3 **Rheological Anaysis**

In MIM, the rheological properties are important as it concerns with the flow ability and the uniform filling of the feedstock during injection moulding. The rheological properties of feedstock can be identified based on the feedstock viscosity, shear sensitivity and temperature sensitivity. The typical value of shear rates for MIM is encountered at the mould range from 100 to 1400 s^{-1} and acceptable value for viscosities is in the range of 100 to 1000 Pa.s[10]. Hence most of rheological studies are perform in this range of viscosity.



(a) (b)

Figure 4 (a) : The relation between viscosity of feedstock and shear rate (b) : The relation between viscosity of feedstock and shear rate

Figure 4(a) and (b) depict the effects of shear rate and temperature on the viscosity of the feedstock which exhibits a pseudoplastic or shear thinning flow behaviour. These findings suggest that the composite binder comprising of waste rubber works successfully with M2 High Speed Steel to produce a feedstock which is favourable for injection moulding process.

5.6.4 Injection Moulding

An optimized powder loading of 65 vol % proved suitable to produced defects free M2 HSS injection moulded part as shown in Fig.5 by using injection pressure of 400 bar and mold temperature of 230°C.

Figure 5: Injection moulded M2 HSS tensile test parts

5.6.5 Green part characterization

Fig. 6 (a) and (b) show scanning electron micrograph of the fractured surface and surface of the green part respectively exhibit that the M2 HSS particles were enwrapped by the binders equably and ensuring the density stability of the green parts. It also appeared that there are some voids between the metal particles owing to shrinkage of the binder that occurred during cooling.

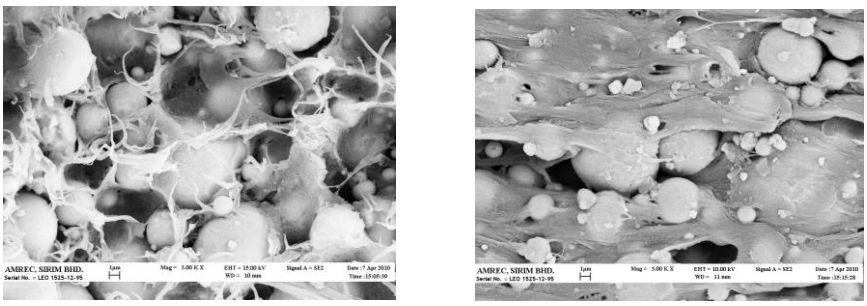


Figure 6. Scanning electron micrograph of (a) fractured surface of the green body (b) surface of the green body.

5.6.6 Debinding

In order to reduce the total debinding time, solvent debinding was conducted by immersing the parts in a bath with n-heptane where PS and SA are soluble.

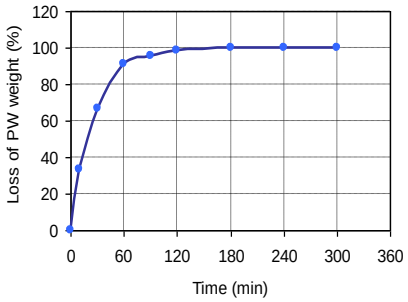


Figure 8. Correlation of binder removal efficiency and debinding time.

Figure 8 displays the correlation of binder removal efficiency with debinding time (1–5 h) at a temperature of 60 °C which indicates reduction of debinding rate with increasing debinding time. It has been observed that the part achieved 90% binder removal after immersion in n-heptane for duration of 120 minutes without defects such as cracking, slumping and sagging.

Different positions, including the fractured surface and the outer part of the as-molded parts were examined by using Scanning Electron Microscopy (SEM) in order to observe the binder distribution porosities resulted from the leaching process.

Fig.9 (a) and (b) show scanning electron micrograph of the fractured surface of the injection moulded parts which have been leached for 60 minutes and 120 minutes respectively.

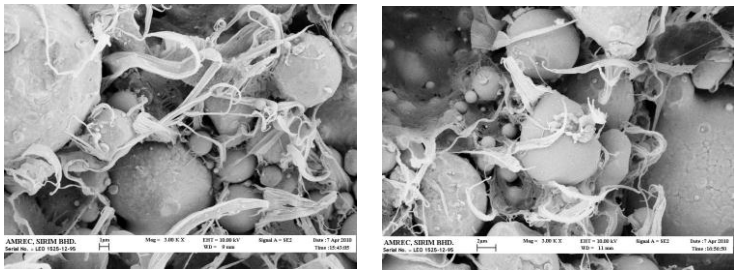


Figure 9: Scanning electron micrograph of fractured surface of (a) leached for 60 minutes (b) leached for 120 minutes

A SEM micrograph of solvent leached part Fig. 9 (a) clearly illustrates the open pore channels formed after partial removal of PW and SA. These channels allowed more rapid removal of the remaining binder, without cracking, blistering or general swelling during subsequent sintering stage. As depicted in Fig. 9(b) a network of polyethylene ligaments remained, binding and holding the M2 HSS particles together to provide the brown part with sufficient brown strength to be handled.

From this study, it shows that solvent debinding process was found to be easier and successful to remove all soluble binders in the as-molded parts of M2 HSS in less than 5 hours. The whole debinding cycle is about 2 hours in duration, which represents a considerable shortening compared with the conventional thermal degradation process which normally lasted for duration of 14 hours.

5.6.7 **Characterization of sintered part**

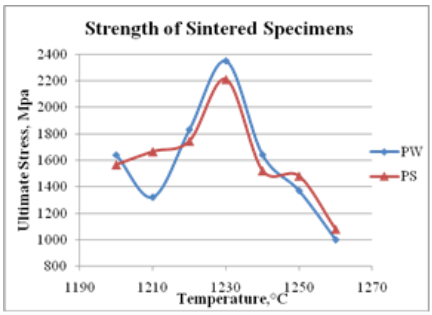


Fig. 10: Effects of sintering temperature on the strength of sintered parts.

Transverse rupture strength (TRS) test was performed according to MPIF 41. The results obtained suggest that the strength sintered parts for both PW and PS binder system have the same pattern in which the value of ultimate stress increases rapidly correlative with sintering temperature up to 1230°C to build a sharp peak, then drastically reduce, which is believed due to a very narrow sintering window of M2 HSS. As depicted in Figure 10, the part sintered at a temperature of 1230°C had resulted in favorable effect and possessed maximum yield stress of 2351MPa and 2210MPa for PW and PS respectively, presumably due to enhancement in density and reduction in porosities of the sintered part.

At sintering temperature higher than 1230°C, the ultimate stress rapidly drop due to grain growth phenomena, which is associated with the development of discontinuous carbide films and the appearance of brittle eutectic carbide phase at the prior austenite grains. The microstructure changes of the sintered parts therefore lowered the ultimate stress and deteriorated the mechanical properties of the sintered parts [9-14].

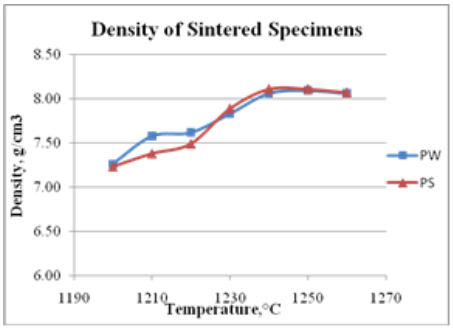


Fig. 11: Effects of sintering temperature on the density of sintered parts.

Density evaluation for sintered parts PW and PS revealed that densification of the sintered part took place when the part was sintered at temperature ranging from 1220°C to 1240°C. Densification rate achieved at sintering temperature below 1220°C was consistent with solid state diffusion and the sintered density obtained was below the theoretical density, which is 7.97g/cm^3. It is interesting to note that once the liquid phase began to appear, the densification rate became very rapid, resulting in sintered density higher than the theoretical density [13-14].
Rapid densification that appears in the sintered part was believed due to super-solidus liquid phase sintering [13-14,19]. It has been observed that part sintered at sintering temperature of 1230°C attained near full density while those sintered at higher sintering temperature possessed sintered density which was

greater than the theoretical density. Liu et al.(2000) discussed that specimens sintered above a sintering temperature of 1210°C had attained near full density as a result of rapid densification. As the metal powder used in this work is coarser, it is expected that the near full density was achieved at higher sintering temperature [6,7].

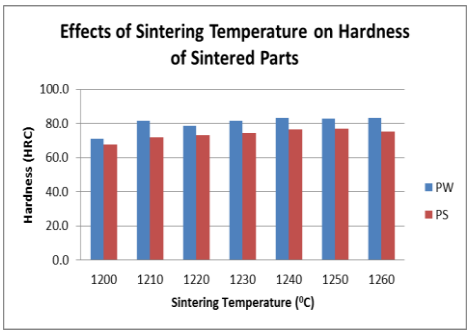
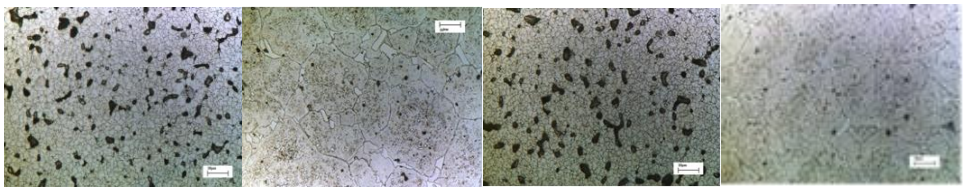


Fig. 12: Effects of sintering temperature on the hardness of sintered parts

Rapid densification due to super-solidus liquid phase sintering that took place at sintering temperature greater than 1230°C accounts for the high hardness value of both binder systems as depicted in Figure 12. The hardness values of sintered part from both binder systems increase with increment in the sintering temperature, confirmed the densification rate which has been discussed earlier.

Figure 13(a), (b), (c) and (d) depict the microstructure of M2 HSS PW and PS parts which were sintered at a temperature of 1200°C, and 1250°C respectively. The micrograph for both sintered parts from both binder systems clearly showed similar correlation of microstructure with the sintering temperature. At sintering temperature of 1200°C, the microstructure possessed small grains and a large number of irregular shape large pores spread inside the grain boundary as the sintered specimen was not sufficiently densified yet [13-14].



(a) (b) (c) (d)

Fig.13 : Microstructure of (a) PW part sintered at 1200°C, (b) PW part sintered at 1250°C, (a) PS part sintered at 1200°C, (b) PS part sintered at 1250°C

As the sintering temperature increases to 1230°C, the grain size increased slightly and the pores volume shrunk. At this stage, the sintered part had attained near full density. At sintering temperature range of 1240°C to 1250°C, angular carbides in the grain boundaries gradually showed grain growth with the increasing sintering temperature whilst the small carbide inside the grain shrunk and reduced.

The fracture surface of PW and PS specimens sintered at different temperature were observed by SEM. Figure 14 (a) and (b) display fracture surface of PW and PS specimens which were sintered at a temperature of 1210° showed the presence of large pores between the particles. The micrograph also clearly indicated partial interconnection of the particles which had taken place since the original form of the powder can still be observed.

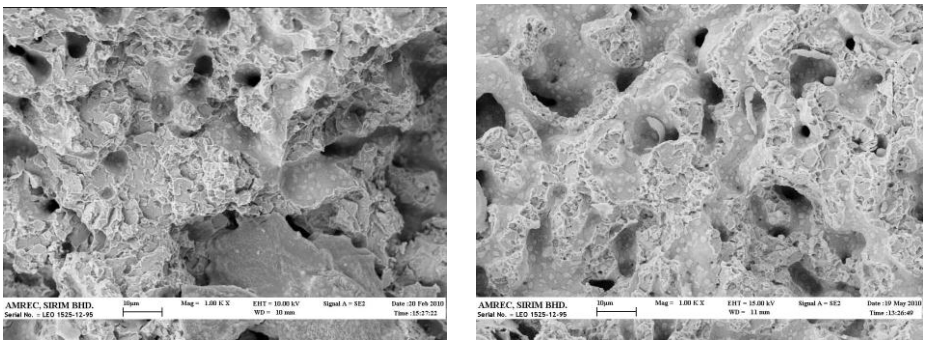


Fig.14 : Fracture morphologies of of (a) PW part sintered at 1210°C, (b) PS part sintered at 1210°C

However, as the sintering temperature is increased to 1240°C, near full density is achieved as the grain boundaries appeared to replace the powder boundary and the grain begin to grow. At a sintering temperature range of 1240°C to 1260°C, the microstructure begins to coarsen and the micrograph indicate that the grain size increases as the sintering temperature was raised from 1240°C to 1260°C. However, as the sintering was raised to 1260°C, the sintered specimen was found to possess inter-grain-boundary fracture characteristic as shown in Figure 15 (a) and (b) [5-9]. According to Liu et al., deposition of more brittle carbide along the grains boundaries could be the main source for the crack formation [13-14].

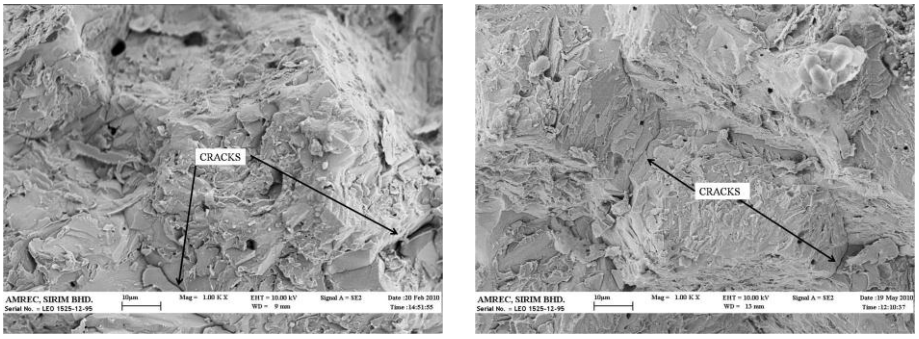


Fig.15 : Fracture morphologies of of (a) PW part sintered at 1260°C, (b) PS part sintered at 1260°C