UNIVERSITI PUTRA MALAYSIA

INJECTION MOLDING OF STAINLESS STEEL (SUS 3 16L) POWDER USING A THERMOPLASTIC BINDER SYSTEM

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FSAS 2002 34

INJECTION MOLDING OF STAINLESS STEEL (SUS 316L) POWDER USING A THERMOPLASTIC BINDER SYSTEM

By

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

September 2002

Abstract of the thesis submitted to the Senate of Universiti Putra Malaysia in fulfillment of the requirements for the degree of Doctor of Philosophy

INJECTION MOLDING OF STAINLESS STEEL (SUS 316L) POWDER USING A THERMOPLASTIC BINDER SYSTEM

By

ROSDI IBRAHIM

September 2002

Chairman : **Prof. Mohd Yusof Sulaiman, Ph.D.**

Faculty : **Science and Environmental Studies**

The thesis presents the results of the investigation carried out to determine the effect of injection pressure and temperature, sintering temperature, sintering atmosphere, volume fraction of powder/binder mixture and composition of the binder system on the properties of 316L stainless steel specimens produced by the Metal Injection Molding (MIM) technique. The stainless steel powder of 16 μm average particle size was mixed with a thermoplastic binder comprising polyethylene, paraffin wax and stearic acid in the ratios of 35/60/5, 30/60/10 and 25/60/15 respectively using a z-blade mixer at the temperature of 160°C. The volume fractions of the powder used in the investigation were 65% and 58%. The specimen of tensile test shape was molded using an 80-ton Arburg injection molding machine at various injection pressures and temperatures. Each

molded specimen was debinded at the rate of 0.2°C/min until the temperature of 440°C using the thermal debinding method. For the hydrogen atmosphere the debinded specimen was sintered at the following temperatures: 1050°C, 1100°C, 1150°C, 1175°C, 1200°C, 1225°C and 1250°C. In the case of argon and vacuum atmospheres, the debinded specimen was only sintered at 1225°C. The results showed that, as the injection pressure of the injection molding machine increased, the density (7.32g/cm^3), hardness (154.53Hv), ultimate tensile strength (522.83MPa), elongation (8.36%), weight loss (5.31%) and shrinkage (12.47%) of the sintered specimen also increased. But, the porosity level (0.90%) of the sintered specimen decreased. However, as the temperature of the injection molding machine increased, the density (7.24g/cm^3), hardness (148.15Hv), ultimate tensile strength (507.97MPa), elongation (6.69%), weight loss (5.02%) and shrinkage (9.79%) of the sintered specimen decreased. The porosity level (1.56%) of the sintered specimen on the other hand increased. The sintered specimen also exhibited almost complete formation of grain boundaries as sintering progressed resulting in grain growth and spheroidization of the pores. These were the primary factors that contributed to the strength of the sintered specimen.

The effect of sintering under different temperatures showed that, as the temperature of the sintered specimen increased, the density (7.32g/cm^3), hardness (154.53Hv), ultimate tensile strength (522.83MPa), elongation (8.36%), weight loss (5.31%) and shrinkage (12.47%) of the sintered specimen increased. However, the porosity (0.90%) decreased. At the temperatures between 1050 and 1175°C, the interaction between particles could not form a bond. As the temperature of the sintered

specimen increased, the interaction of the particles produced bonding and this resulted in the specimen exhibiting plasticity effect.

The results on the effect of variation of the atmosphere showed that, the density (H_2-7.32g/cm^3, Ar_2-6.99g/cm^3 and Vacuum-7.05g/cm^3) of the specimen sintered under the atmosphere of hydrogen could be accepted as the MIM standard density sintered specimen. On the other hand specimens sintered under hydrogen, argon and vacuum could be accepted as the MIM standard reference for hardness (H_2-154.53Hv, Ar_2-135.47Hv and Vacuum-137.88Hv) and porosity (H_2-0.90%, Ar_2-1.24% and Vacuum-1.09%). The elongation (H_2-8.36%, Ar_2-9.73% and Vacuum-16.79%) of the sintered specimen under vacuum had the highest value, while the sintered specimen under hydrogen had the lowest elongation. Sintered specimen under argon atmosphere had an intermediate elongation. As for the weight loss (H_2-5.31%, Ar_2-5.17% and Vacuum-5.69%), the specimen sintered in argon had the lowest value followed by an increase in value for the specimen sintered in hydrogen and vacuum respectively. In general, the sintered specimen under hydrogen atmosphere showed the highest shrinkage (H_2-12.47%, Ar_2-8.76% and Vacuum-9.48%) while the specimen sintered under argon atmosphere showed the least. The specimen sintered in vacuum exhibited intermediate shrinkage. When the formation of grain boundaries of the specimen sintered under hydrogen atmosphere reached completion, the grain growth and spheroidization of the pores occurred. This contributed to the high strength of the sintered specimen. For the specimen sintered under argon atmosphere, the carbon from the powder or residual binder would react with oxide elements to form carbon monoxide. As the temperature of the sintering process increased, the formation of elemental oxides occurred and the

formation of grain boundaries was not complete. This resulted in the low strength of the sintered specimen. The sintered specimen under vacuum atmosphere experienced a process similar to that involving the argon atmosphere. However, the number of oxygen molecules present was much lower. Therefore, the interaction between the particles increased and, owing to a cleaner interparticle surface among the interacting particles, would contribute to the formation of a high-strength and ductile specimen.

The results on the effect of volume fraction of powder showed that, the sintered specimen A-2 (volume fraction of 65%) had 96.37% of the MIM standard density of 7.60g/cm^3, 0.90% of porosity and 95.58% of ultimate tensile strength of that specified by Metal Powder Industries Federation (MPIF) 35. Besides, the sintered specimen A-2 also had the highest value of elongation (8.36%), weight loss (5.31%) and shrinkage (12.47%) due to the strong interparticle bonding, strong capillary extraction of the binder from the specimen and a high volume fraction of powder employed for the specimen. The density of the sintered specimen A-2 could be accepted for the MIM standard 316L stainless steel sintered specimen according to MPIF 35. Meanwhile, the hardnesses of the sintered specimen A-1 (162.80Hv) and A-2 (154.53Hv) were acceptable within the MIM standard MPIF 35. Micrographs of A-1 showed that the particle surface was not clean enough. Therefore, the interparticle bonding was not strong and this resulted in the low strength of the sintered specimen. On the other hand, micrographs of A-2 showed a clean particle surface indicating a strong interparticle bonding and this led to homogenization and high strength of the sintered specimen.

The results on the effect of binder composition of the binder system showed that the densities of the sintered specimen A (7.15g/cm^3), B (6.87g/cm^3) and C (7.10g/cm^3) were not acceptable within the MIM standard for 316L stainless steel sintered specimens. The value of the hardness (A-189.80Hv, B-183.00Hv and C-162.80Hv) was within the acceptable limits of the MIM standard 35. As the binder's polyethylene content increased from 25% to 35%, the ultimate tensile strength (A-401.30MPa, B-432.80MPa and C-452.70MPa) and shrinkage (A-10.46%, B-11.47% and C-10.47%) of the sintered specimen increased. There were small changes on the elongation (A-4.23%, B-7.39% and C-6.93%) and weight loss (A-9.09%, B-7.71% and C-9.10%) of these sintered specimens. From the observation of micrograph, there are a lot of pores in the grain of the sintered specimen A, B and C. This, resulted in the low strength of these sintered specimens.

As a suggestion for future work, the sintering temperature should be increased to 1380^{o}C and the properties studied. At this temperature, the vacuum should be maintained at 10^{-6} torr to avoid oxidation. The dew point should also be controlled at this temperature.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGACUAN SUNTIKAN SERBUK KELULI KALIS KARAT (SUS 316L) DENGAN MENGGUNAKAN SISTEM PENGIKAT TERMOPLASTIK

Oleh

ROSDI IBRAHIM

September 2002

Pengerusi : **Prof. Mohd. Yusof Sulaiman, Ph.D.**

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Tesis ini mempersembahkan hasil kajian bagi menentukan kesan tekanan suntikan dan suhu, suhu pemanasan, atmosfera pemanasan, pecahan isipadu dalam percampuran serbuk/pengikat dan komposisi sistem pengikat keatas ciri-ciri spesimen keluli kalis karat 316L yang dihasilkan melalui teknik Pengacuan Suntikan Logam (PSL). Serbuk keluli kalis karat dengan purata saiz 16 μm dicampurkan dengan sistem pengikat termoplastik yang terdiri dari polietilina, lilin paraffin dan asid stearik pada nisbah yang berbeza iaitu 35/60/5, 30/60/10 dan 25/60/15 dengan menggunakan alat percampuran bilah-z pada suhu 160°C. Pecahan isipadu serbuk yang digunakan dalam kajian ini adalah pada 65% dan 58%. Spesimen kemudiannya dibentuk dengan menggunakan mesin pengacuan suntikan berjenama Arburg 80-ton pada tekanan

suntikan dan suhu yang berbeza. Setiap spesimen yang telah dibentuk kemudiannya dinyahikatan pada kadar 0.2°C/min hingga suhu 440°C dengan menggunakan kaedah nyahikatan terma. Spesimen yang telah dinyahikatan kemudiannya dipanaskan dibawah atmosfera hidrogen pada suhu berikut 1050°C, 1100°C, 1150°C, 1175°C, 1200°C, 1225°C dan 1250°C. Manakala, bagi kes atmosfera argon dan vakum pula, spesimen yang telah dinyahikatan dipanaskan hanya pada suhu 1225°C. Keputusan menunjukkan bahawa, sekiranya tekanan suntikan pada mesin pengacuan suntikan logam dinaikkan, ini akan menyebabkan ketumpatan (7.32g/cm^3), kekerasan (154.53Hv), kekuatan (522.83MPa), pemanjangan (8.36%), kehilangan berat (5.31%) dan pengecutan (12.47%) pada spesimen yang dipanaskan juga akan meningkat. Sebaliknya, ini akan menyebabkan tahap porositi (0.90%) spesimen yang dipanaskan pula akan berkurangan. Walau bagaimanapun, sekiranya suhu mesin pengacuan suntikan logam dinaikkan, ini akan menyebabkan ketumpatan (7.24g/cm^3), kekerasan (148.15Hv), kekuatan (507.97MPa), pemanjangan (6.69%), kehilangan berat (5.02%) dan pengecutan (9.79%) pada spesimen yang dipanaskan akan berkurangan. Sebaliknya, ini juga akan menyebabkan tahap porositi (1.56%) spesimen yang dipanaskan akan meningkat. Pembentukan lengkap butiran sempadan yang terbentuk pada spesimen yang dipanaskan dan seterusnya, ini akan menyebabkan pengembangan butiran dan liang-liang berbentuk sferoid terhasil. Ini merupakan faktor utama menyumbangkan kepada kekuatan spesimen yang dipanaskan.

Kesan suhu pensinteran yang berbeza menunjukkan bahawa, apabila suhu spesimen yang dipanaskan dinaikkan, ini akan menyebabkan ketumpatan (7.32g/cm^3), kekerasan (154.53Hv), kekuatan (522.83MPa), pemanjangan (8.36%), kehilangan berat

(5.31%) dan pengecutan (12.47%) spesimen yang dipanaskan turut meningkat. Bagaimanapun, porositi (0.90%) pula akan berkurangan. Pada suhu diantara 1050°C hingga 1175°C, didapati interaksi diantara partikel tidak dapat menghasilkan ikatan. Akan tetapi apabila suhu dinaikkan lagi, ini akan meyebabkan interaksi diantara partikel terhasil dan ini menyebabkan kesan keplastikan.

Keputusan keatas kesan atmosfera yang berbeza menunjukkan bahawa, ketumpatan (H_2-7.32g/cm^3, Ar_2-6.99g/cm^3 dan Vacuum-7.05g/cm^3) spesimen yang dipanaskan dibawah atmosfera hidrogen dapat diterima didalam piawaian PSL. Begitu juga, spesimen yang dipanaskan dibawah hidrogen, argon dan vakum juga dapat diterima jika dibandingkan dengan MPIF 35 bagi kekerasan (H_2-154.53Hv, Ar_2-135.47Hv dan Vacuum-137.88Hv) dan porositi (H_2-0.90%, Ar_2-1.24% dan Vacuum-1.09%). Nilai pemanjangan (H_2-8.36%, Ar_2-9.73% dan Vacuum-16.79%) spesimen yang dipanaskan dibawah atmosfera vakum didapati mempunyai nilai yang tertinggi, manakala spesimen yang dipanaskan dibawah atmosfera hidrogen mempunyai nilai pemanjangan yang paling rendah. Sementara itu, spesimen yang dipanaskan dibawah atmosfera argon mempunyai nilai dipertengahan. Begitu juga bagi nilai kehilangan berat (H_2-5.31%, Ar_2-5.17% dan Vacuum-5.69%), spesimen yang dipanaskan dibawah atmosfera argon mempunyai nilai yang terendah dan kehilangan berat meningkat bagi spesimen yang dipanaskan dibawah atmosfera hidrogen dan vakum. Secara umumnya, spesimen yang dipanaskan dibawah atmosfera hidrogen menunjukkan nilai pengecutan (H_2-12.47%, Ar_2-8.76% dan Vacuum-9.48%) yang paling tinggi. Manakala, spesimen yang dipanaskan dibawah atmosfera argon mempunyai nilai yang paling rendah. Spesimen yang dipanaskan dibawah atmosfera vakum mempunyai nilai pengecutan

dipertengahan. Apabila pembentukan butiran sempadan pada spesimen yang dipanaskan dibawah atmosfera hidrogen telah lengkap, ini akan menyebabkan pertumbuhan butiran dan liang berbentuk sferoid terhasil. Ini menyumbang kepada peningkatan kekuatan spesimen yang dipanaskan. Bagi spesimen yang dipanaskan dibawah atmosfera argon, didapati karbon dari serbuk atau lebihan pengikat akan berinteraksi dengan oksigen untuk membentuk karbon monoksida. Sekiranya suhu pemanasan dinaikkan, ini akan menyebabkan pembentukan elemen oksida dan pembentukan butiran sempadan tidak lengkap. Ini turut menyebabkan kekuatan spesimen yang dipanaskan menjadi lemah. Spesimen yang dipanaskan dibawah atmosfera vakum pula didapati mempunyai proses yang sama dengan spesimen yang dipanaskan dibawah atmosfera argon. Walau bagaimanapun, kandungan oksigennya lebih rendah. Dengan itu, menyebabkan interaksi diantara partikel dan permukaan partikel menjadi lebih bersih dan ini turut menyumbang kepada peningkatan kekuatan dan kekenyalan.

Keputusan keatas kesan pecahan isipadu serbuk menunjukkan bahawa, spesimen yang dipanaskan A-2 (pecahan isipadu pada 65 vol.%) mempunyai 96.37% nilai ketumpatan berbanding dengan nilai piawaian MIM ketumpatan iaitu 7.60 g/cm^3, 0.90% nilai porositi dan 95.58% dari kekuatan yang dispesifikasikan oleh Persekutuan Industri Serbuk Logam (MPIF) 35. Selain daripada itu, spesimen yang dipanaskan A-2 juga mempunyai nilai pemanjangan (8.36%), kehilangan berat (5.31%) dan pengecutan (12.47%) yang tertinggi disebabkan oleh kekuatan ikatan diantara partikel, penyaringan rerambut yang kuat terhadap pengikat dari spesimen dan penggunaan pecahan isipadu serbuk yang tinggi pada spesimen. Ketumpatan spesimen yang dipanaskan A-2 boleh diterima untuk piawaian PSL bagi keluli kalis karat 316L merujuk kepada MPIF 35.

Sementara itu, kekerasan spesimen yang dipanaskan A-1 (162.80Hv) dan A-2 (154.53Hv) didapati boleh diterima didalam MPIF 35. Mikrostruktur A-1 menunjukkan bahawa, permukaan partikel didapati tidak berapa bersih. Dengan itu, ianya akan menyebabkan ikatan diantara partikel tidak kuat dan ini menyumbang kepada kelemahan pada spesimen yang dipanaskan. Sebaliknya, mikrostruktur A-2 menunjukkan permukaan partikelnya yang bersih dan ini menyumbang kepada ikatan diantara partikel yang kuat dan ianya juga turut menunjukkan keseragaman dan kekuatan yang tinggi pada spesimen yang dipanaskan.

Keputusan keatas kesan komposisi pada sistem pengikat menunjukkan bahawa, ketumpatan spesimen yang dipanaskan A (7.15g/cm^3), B (6.87g/cm^3) dan C (7.10g/cm^3) tidak dapat diterima didalam julat piawaian PSL bagi keluli kalis karat 316L . Manakala, nilai kekerasan (A-189.80Hv, B-183.00Hv dan C-162.80Hv) pula didapati boleh diterima jika dibandingkan dengan MPIF 35. Apabila komposisi pengikat iaitu polietilina dinaikan dari 25% kepada 35%, didapati kekuatan (A-401.30MPa, B-432.80MPa dan C-452.70MPa) dan pengecutan (A-10.46%, B-11.47% dan C-10.47%) spesimen yang dipanaskan juga akan meningkat. Akan tetapi, nilai pemanjangan (A-4.23%, B-7.39% dan C-6.93%) dan kehilangan berat (A-9.09%, B-7.71% dan C-9.10%) bagi spesimen yang dipanaskan tidak berubah dengan banyaknya. Spesimen tersinter A, B dan C didapati mempunyai lompang-lompang halus tertabur didalam butiran-butirannya. Hal ini menyebabkan spesimen tersinter tersebut tidak kuat.

Cadangan untuk penyelidikan yang akan datang adalah, suhu pemanasan perlu dinaikkan lagi sehingga pada suhu 1380^{o}C dan penciriannya perlu dikaji. Pada suhu

tersebut, tahap vakum perlu ditingkatkan lagi sehingga 10^{-6} torr untuk mengelak dari berlakunya pengoksidaan. Pengawalan titik dew perlu diberi perhatian apabila spesimen dipanaskan pada suhu tersebut.

ACKNOWLEDGEMENTS

In the name of Allah, Most Gracious, Most Merciful

Be all for the Almighty Allah for giving me the utmost strength and courage to complete this project successfully.

It gives me much pleasure to acknowledge and thank many individuals, institutions and SIRIM Berhad for their significant contributions during the entire course of this study.

I would like to express my utmost thanks, gratitude and appreciation to my chairman, Prof. Dr. Mohd. Yusof Sulaiman for his original idea, helpful, advice, invaluable guidance, suggestion, encouragement, stimulating discussion and patience throughout this study.

I am particularly happy with and appreciate his approachable manner which made this exercise a pleasant one.

Similar appreciation is extended to members of my supervisory committee, Prof. Dr. Abdul Halim Shaari, Prof. Dr. Kaida Khalid and Assoc. Prof. Dr. Zainal Abidin Sulaiman for their constructive comments on the work. Also special thank to Dr. Azmi Idris, Senior General Manager, Advanced Materials Research Center (AMREC), SIRIM Berhad for his moral support and valuable ideas.

Field and laboratory works were assisted by research staff of the Powder Metallurgy Group, Advanced Materials Research Center (AMREC), SIRIM Berhad. They are En. Hamdan Mohamad and En. Ahmad Sabata. Their earnestness and hardwork were the decisive factor in making this study a reality.

Last but not least, heartfelt appreciation and love are due to my father, En. Ibrahim Sham, my brothers, sisters and friends especially to En. Shamsul Muhamad (Institute Medical Research). I wish them every success in this world and hereafter under the guidance and in the path of Allah s.w.t. Wassalam.

I certify that an Examination Committee met on 19th September 2002 to conduct the final examination of Rosdi Ibrahim on his Doctor of Philosophy thesis entitled "Injection Molding of Stainless Steel (SUS 316L) Powder Using A Thermoplastic Binder System" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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This thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee are as follows:

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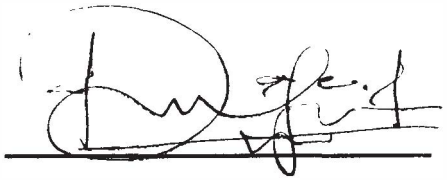
Date:

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have duly acknowledge. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.



ROSDI IBRAHIM

DATE:

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