Ceramic dies selection for electrical resistance sintering of metallic materials

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ABSTRACT. Processing metallic powders by electrical resistance sintering requires the use of insulating ceramics dies. Selecting the appropriate ceramic material according to the electrical, thermal and mechanical properties is a need. Dies produced with several ceramic materials have been tested during the production of cemented carbide in order to check their behaviour in the process and final product properties. Tialite/mullite, zircon/mullite, zirconium phosphate based ceramic, yttria-stabilized zirconia and sialon, in most cases with modified compositions and shaping processes in order to achieve a high density, have been tested. Dry powder processing by cold isostatic pressing and furnace sintering resulted to be the better process for dies production. The effect of die properties on the produced cemented carbide, and the behaviour and life of the die during the production have been analysed. Very smooth die surface increases the number of cycles withstood during metallic parts production, because of lower extraction stresses, as checked for sialon dies. Zirconium phosphate based dies, with low thermal conductivity, show the most densified hard metal parts surface.

KEYWORDS: ceramic materials; insulating dies; electrical resistance sintering; cemented carbide

1. INTRODUCTION

Appropriate materials selection is a key aspect for any engineering application [1-3]. Improvements in the properties of the selected material usually lead to significant economic savings during the use of the produced parts. This material selection does not only apply to the final products, but also to the different materials involved in their production. Thus, when the equipment necessary to obtain the final product is susceptible of being a problems source, its adequate selection can be as critical as the properties of the final product itself. One such case is the fabrication of cemented carbides.

Hard metal or cemented carbide is the name given to a mixture of tungsten carbide (WC) and a binder metal as cobalt (Co) [4-5], although other compositions are also possible. The resulting composite, with composition usually tailored to the desired application, is a very hard material used for the production of cutting tools, as drills, saws or inserts, needed for processing metallic materials, or geo-engineering [6]. The resulting product shows, in general, better global behaviour than other options as high-speed steels.

The production of these cemented carbides is usually carried out by a compaction process into a specific form, a green part, that is then furnace sintered to obtain the final part. During sintering, the binder partially melts, surrounding the carbides that remain in solid state at the sintering temperatures. The obtained metal matrix composite is additionally grinded and polished to the final shape [7].

A new technique for the production of cemented carbides, based on sintering the pressed powders mixture by leading an electrical current through it (Joule effect), has been recently studied [8]. This technique, known as electrical resistance sintering (ERS), is one of the modalities of the known as Field Assisted Sintering Techniques [9], having the possibility of restraining grain growth and therefore improving properties. During ERS, powders are placed into an isolating die, and two conductive electrodes/punches act to exert the desired pressure to compact the powders, at the time that a high intensity current, of up to approximately 15 kA, with a low voltage of up to 10 V, passes through the powder mass. These features are easily satisfied by adapting a spot welding machine to carry out the ERS process [10, 11].

A very important factor that affects the electrical consolidation is the selection of the die material. The die material has to be good electrical and thermal insulator, being resistant to thermal shocks, chemically inert, and having good mechanical and wear properties. This will make the electric current passing through the metallic powders, avoiding heat leaks and a more efficient heating and sintering of the cemented carbides. In addition, having the adequate roughness for de-moulding will decrease wear, and a good mechanical resistance will help resisting stresses. This makes the selection of the appropriate die material a key point in the development of this novel cemented carbides processing. Requirements for the ceramic dies were set based on bibliography and previous experience as follows: flexural strength higher than 50 MPa, thermal expansion lower than $4 \cdot 10^{-6}$ 1/°C, electrical resistivity higher than 10^7 $\Omega \cdot$m and refractoriness (temperatures higher than 1000 °C).

According to the literature [12-31], the following refractory oxide materials with attractive properties can be considered: aluminium titanate or tialite (Al_2TiO_5), sodium zirconium phosphate NZP based ceramics, mullite ($3Al_2O_3 \cdot 2SiO_2$), aluminum oxide (Al_2O_3), zirconium oxide or zirconia (ZrO_2), zirconium silicate or zircon ($ZrSiO_4$), cordierite ($2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$), silicon nitride ($Si_3N_4$), boron nitride-zirconium oxide composites (BN-ZrO_2) and zircon-mullite ($ZrSiO_4$-$3Al_2O_3 \cdot 2SiO_2$). These advanced ceramics exhibit excellent properties and are used in high temperature and very demanding applications: metallurgical industry, glass industry, electronics, thermal barrier coatings and thermal shock resistant, high temperature fuel cells, oxygen detectors, catalysts supports, wall flow filters for diesel particulate emission control in automotive industry, etc. However, alumina does not withstand thermal shocks, and cordierite has a maximum service temperature close to 1000 °C, therefore not being considered for the current application. The materials with properties fulfilling most of the high demanding requirements set above are mainly: modified aluminum titanate-mullite composites, NZP ceramics, mullite, zirconia, silicon nitride and zircon-mullite composites.

In this paper, several ceramic materials have been tested in order to establish their benefits and drawbacks in the ERS process. Some of these ceramics have been specifically prepared for this application, whereas other ones are commercial ceramics. Among the many possible available compositions and production techniques, an important goal was always considered during dies production. Taking into account that the durability of the ceramic dies is limited, relatively cheap materials and production techniques were selected. Therefore, considering some basic properties of candidate ceramics, several different ceramics and production techniques have been tested.

2. EXPERIMENTAL PROCEDURE and MATERIALS

Several different ceramic dies, produced from custom or commercial powders, as well as net-shape acquired dies, have been tested in this work. Criteria called for in the selection of those candidate materials are low electrical and thermal conductivity, thermal shock resistance, high hardness and toughness, high density giving way to low-roughness surface finish, etc. Three of the tested powdered materials have been synthesized in this work, i.e., modified tialite/mullite ($Al_{1.6}Fe_{0.4}TiO_5$-$3Al_2O_3{\cdot}2SiO_2$) AT/M, modified zircon/mullite (ZrO_2-$3Al_2O_3{\cdot}2SiO_2$-MgO) Z/M, and modified sodium zirconium phosphate based ceramics ($Ba_{1.22}Zr_4P_{5.56}Si_{0.44}O_{24}$-ZnO) NZP. Commercial powder used was yttria-stabilized zirconia (ZrO_2-8 mol% Y_2O_3) 8YSZ. In all these cases, the use of fine powder fractions and of various sintering agents were tested in order to obtain low porosity samples.

Regarding die shaping methods suitable for the production of large and complicated ceramic parts, various multi-step solid state reaction processes were tested with the new developed and commercial powders, i.e., slip casting, extrusion, uniaxial pressing (UP) and cold isostatic pressing (CIP).

The optimum manufacturing process for each ceramic was selected and optimization of the manufacturing parameters (e.g. pressurizing load, viscosity, solids load, sintering temperature) was carried out. Dies were characterized regarding to the following key properties: porosity, coefficient of thermal expansion (CTE), electrical resistivity and thermal conductivity.

Finally, as benchmarking, ready to use β-SiAlON (Si_3N_4-Al_2O_3-Y_2O_3) dies were acquired and tested.

The optimum ERS cycle (temperature and time) was used in order to obtain samples with the minimum possible porosity and the combination of the properties described above.

2.1. DIES PRODUCTION. Taking into account the aforementioned set of required values, ceramic materials synthesis process and die production process were both optimized for each material. Optimized final ceramics were afterwards tested regarding their effect on ERS. The following four materials were synthesized and shaped.

Modified aluminum titanate-mullite composites: Tialite powder (AT, aluminum titanate) was in-house produced by solid-state reaction, starting from ceramic oxides, while mullite (M) was a commercial powder (Symulox M72, Nabaltec, Germany). After wet mixing, grinding and drying, the resulting modified tialite/mullite (AT/M) is a composite powder with D_{90} = 29.6 µm that can be used for dies production.

Dies were produced by using CIP for shaping the AT/M powders, at 2000 bar for 1 min. After sintering the green dies at 1450 °C for 3 hours, they exhibited a porosity of 6 %, measured by Archimedes method. Sintering at higher temperature, closer to materials melting point, or longer sintering times lead to microcracking, making the material more brittle. Therefore, the above sintering conditions were selected as the optimum for this composite. The addition of sintering agents, e.g. 1wt% ZrO_2 or 0.5%wt SiO_2 [23-24], was examined to further decease the porosity, although no improvement was achieved.

Optimization of the surface quality of the produced AT/M ceramic dies was tried using a finer fraction of the composite powder. It was proved that shaping a fraction with approximately half the value of D_{90} compared to the above (D_{90} = 13.05 µm) results to smother surfaces of the produced dies.

Extrusion was also used for shaping AT/M powders. Several cylindrical samples were produced, and preliminary tests showed that extrusion was satisfactory for the AT/M ceramic composite. In general, extrusion of ceramic powders requires additions of binders, plasticizers and water, in order to produce a plastic extrudable mass. In the present work, additions should be at the lowest percentage in the mix, in order to achieve very dense parts, since organic additives (binders, plasticizers) promote porosity after their removal in the sintering step. Finally, it was decided to focus mainly on dry powder shaping methods, for the different materials.

Modified zircon/mullite composites: Commercial zircon/mullite Z/M powder (Symulox Z72, Nabaltec, Germany), composed of microcrystalline ZrO_2 and $3Al_2O_3 \cdot 2SiO_2$, has a particle size D_{90} = 24.4 µm, outstanding high temperature strength, thermal shock resistance and high chemical and mechanical resistance.

These powders were shaped by CIP at 3000 bar for 1 min and sintered at 1550 °C for 2 h, attaining 80% density. Customized powders by adding various oxides in different amounts [25-27], i.e., zircon, aluminum oxide, zircon and aluminum oxide, mullite and magnesium oxide, were produced. Best results were obtained for 1 wt% MgO addition and 1600 °C, 8 h sintering, leading to a porosity reduction down to 1.5%.

Modified NZP ceramics: A modified sodium zirconium phosphate NZP ceramic with similar structure to sodium zirconium phosphate ($NaZr_2P_3O_{12}$) was also synthetized in a multi-step process [28]. These new NZP powders were shaped by UP at 2000 bar and sintered at 1400 °C for 16 h. Produced parts exhibit very high porosity close to 50%. Therefore, the addition of ZnO as sintering agent was decided [29]. Oxygen vacancies appear in the NZP crystal because of Zr positions substituted for Zn ions with a lower oxidation state. This, in turn, increases the diffusion promoting densification. The modified powders, produced after appropriate mixing, milling and drying, resulted in a size D_{90} = 4.1 µm. Additions of 1 and 2wt% ZnO to the NZP powder were examined. Measured porosity values of the produced samples after sintering were 3.1% and 2.4% respectively, revealing the effective role of ZnO for parts densification.

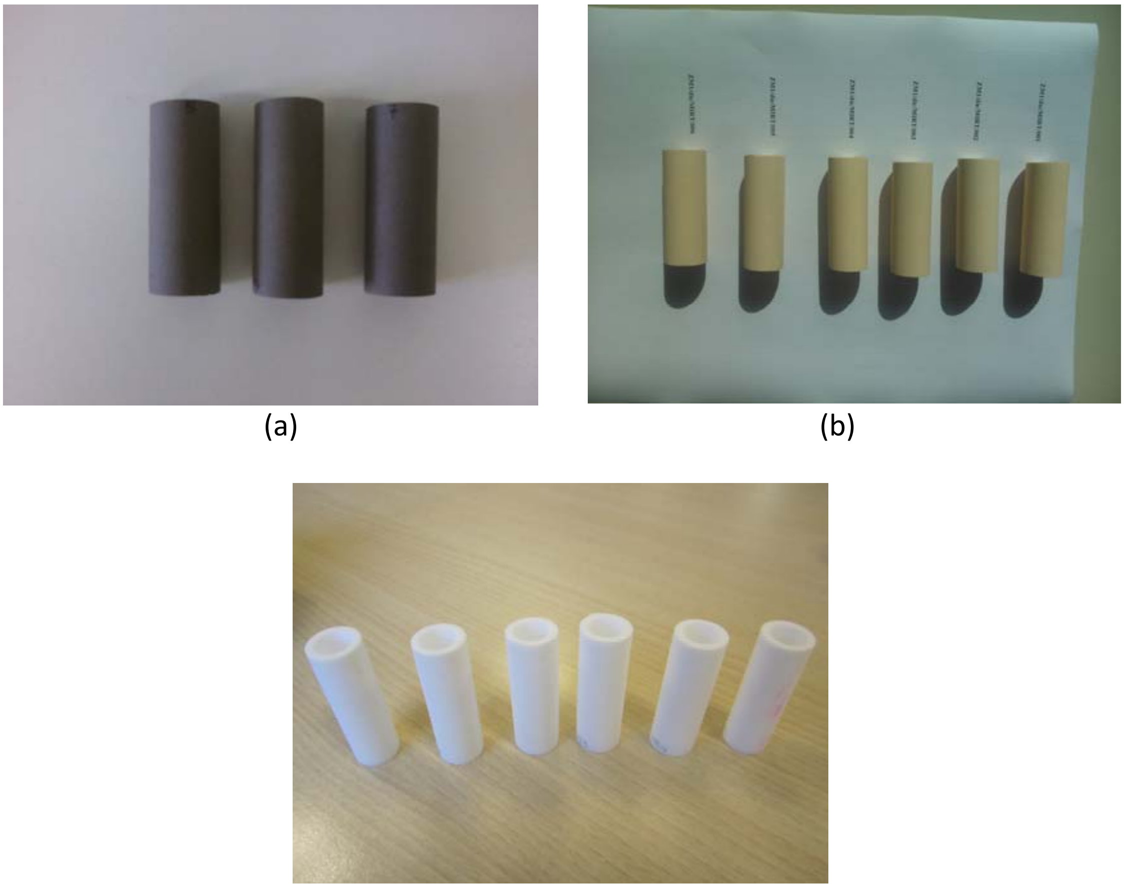
Further decrease of porosity was achieved using the more effective CIP technique (2000 bar for 1 min), reaching a porosity of 1.7 % for NZP samples with 2wt% ZnO, sintered at 1400 °C for 16h.

Stabilized zirconia powders: Regarding commercially available powders, two fully stabilized zirconia powders 8YSZ were used: a raw powder consisting of agglomerates (D_{90} = 19.5 µm), and a spray dried ready to press powder with D_{90} = 2.9 µm for more effective powder compaction and better quality of parts. Both powders were from

Imerys Fused Minerals, Germany. Better results regarding the quality of the produced dies were obtained with the ready to press powder.

The 8YSZ ready to press powder was isostatically pressed at 2000 bar for 1 min. After sintering at 1550 °C for 8 h samples exhibited porosities of 2.1 - 2.3%. Further decrease of porosity, down to 1.7% was achieved after sintering at 1600 °C. The surface quality of these ceramic dies was quite good, with a very smooth finishing. Slip casting was also effective for YSZ powder. However, wall thickness and dimensional accuracy achieved with this technique were not good enough.

Cylindrical ceramic dies of all the previous powders were produced by CIP using a polymeric mould and a metallic mandrel, in the desired dimensions. Dies had D_{in} 12mm, D_{out} 17 mm and various lengths up to 60 mm (Figure 1).



(a) (b)

(c)

Figure 1. Dies produced by CIP and sintering from (a) AT/M, (b) Z/M and (c) NZP powders.

Powder characteristics and conditions applied during die production, as well as the final porosity achieved, are summarized in Table 1.

Table 1. Selected conditions for dies production from ceramic powders and final porosity obtained.

Powder	Particle Size (µm)	Shaping parameters	Sintering	Porosity (%)
AT/M	$D_{90} = 13.05$	CIP, 2000 bar, 1 min	1450 °C, 3 h	6.0
Z/M	$D_{90} = 24.4$	CIP, 3000 bar, 1 min	1600 °C, 8 h	1.5
NZP	$D_{90} = 4.1$	CIP, 2000 bar, 1 min	1400 °C, 16 h	1.7
8YSZ	$D_{90} = 2.9$	CIP, 2000 bar, 1 min	1600 °C, 8 h	1.7

Finally, commercial sialon tubes with the final dimensions of 12 mm inner diameter were acquired (Syalon 050, International Syalons, Newcastle, United Kingdom) to be tested and compared to dies developed at the lab. These materials were acquired with a high quality surface finish to check the difference between machined and not machined surfaces.

2.2. DIES MATERIALS PROPERTIES. Before ERS tests, the thermal shock behavior of the dies, being a key property, was studied. Cylinders were tested by two methods: (1) pouring molten metal at approximately 1400 °C directly in the dies and, (2) quenching the dies from a high temperature (1200 °C) to cold water (20 °C). From both tests, it was revealed that 8YSZ dies do not appropriately withstand severe temperature changes. On the other hand, AT/M composites and NZP ceramics proved to be the best to resist the tests.

Bulk samples were also prepared from modified AT/M, NZP and commercial 8YSZ for additional characterization. The results gathered in Table 2 showed that non-commercial ceramics fulfill most of the high demanding requirements for the ceramic dies. They are excellent thermal and electric insulators, and exhibit very low thermal expansion on heating. From commercially available materials, sialon seems to be an additional suitable candidate.

Table 2. Characterization results of die ceramic materials. (*Based on literature [30-31])

Property	AT/M	NZP	8YSZ	Sialon
Elect. Resist. (Ω·cm)	$1.4 \cdot 10^{10}$ (25 °C)	$6.11 \cdot 10^{14}$ (25 °C)	$1.9 \cdot 10^{13}$ (25 °C)	$1.0 \cdot 10^{10}$ (25 °C)

Thermal Conductivity (W/mK)	2.1 (25 °C) 2.0 (300 °C)	1.1 (25 °C) 1.1 (300 °C)	1.9 (25 °C) 2.0 (300 °C)	25* (25 °C)
CTE (ppm/K)	2.2 (25-100 °C) 3.4 (25-520 °C)	1.6 (25- 90 °C) 1.8 (25-510 °C)	8.3 (25-110 °C) 9.1 (25-510 °C)	3.0* (25-1200 °C)

2.3. ERS TESTING CONDITIONS. All the ceramic dies were tested in ERS experiences with the following conditions: (i) the material to be ERSed was submicron WC-6 wt.% Co cemented carbide, (ii) a pressure of 100 MPa was applied all over the sintering cycle, (iii) current intensity of 7 kA and (iv) dwelling time of 700 ms were used. Mean properties of final 12 mm in diameter, approximately 4 mm height cemented carbide parts were: density 11.86 g/cm^3, hardness 1980 HV$_{30}$ and K$_{IC}$ 9.7 MPa·m$^{1/2}$. Those are values in the typical range for these cemented materials.

The mechanical requirements of the dies, mainly due to the applied pressure, advise to wrap them with a metallic hoop. However, a certain lack of dimensional precision makes this a quite complicated task. As shown in Figure 2, wrapping in a steel tube consisted in inserting a lead foil in between the ceramic and an external 25 mm inner diameter steel tube, then heating slowly up to 450 ºC and cooling slowly, and repeating the process up to filling the cavity. Portland cement was used to close the tubes lower end. Considering dimensions, freezing shrinkage and thermal expansion coefficients, slight compression stresses are produced on the ceramic tubes. Unfortunately, sometimes cement flows up between the die and the steel tube. In such cases, lead did not completely cover dies mouth, leading to stresses increase at the ceramic tube end when extracting the finished part.

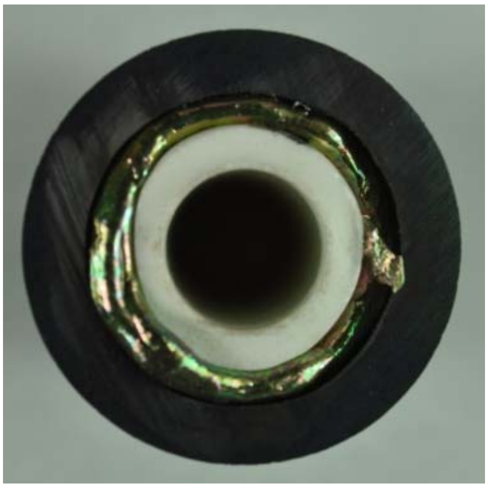


Figure 2. Wrapped ceramic die, with a lead layer and an external steel tube (25 mm inner diameter).

After every ERS experience, several variables were analysed: die roughness, extracting force of the cemented carbide part, and durability of the ceramic die, but also the density and microscopic and mechanical characteristics of the produced cemented carbide.

Regarding roughness, measurements were carried out in the die both initially and after each experiment. According to the Standards [32], various parameters of the roughness profile were studied, average of the roughness profile R_a, maximum profile height R_y, and ten-point mean roughness R_z. A stylus reaching 55 mm maximum from the die mouth was used along with a Mitutoyo HT-12R equipment.

3. RESULTS AND DISCUSSION

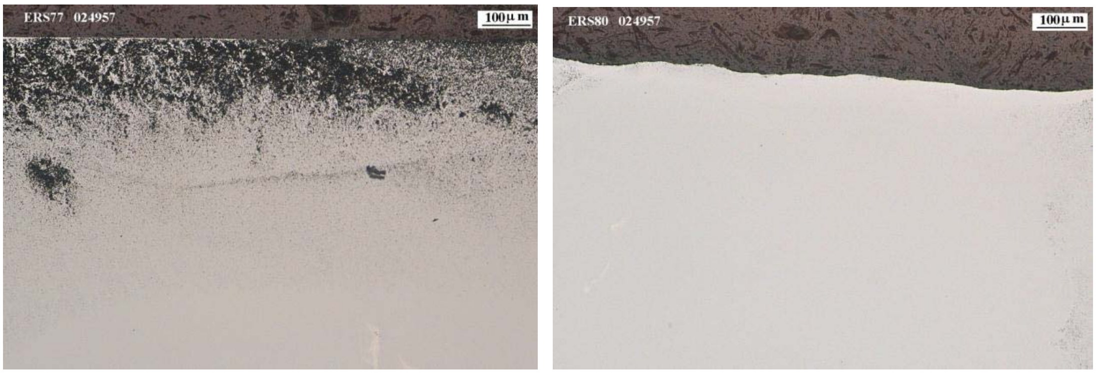
According to the aforementioned procedure, hard metal samples were prepared using the material candidates for the manufacturing of the ceramic dies, that is AT/M, Z/M, NZP, 8YSZ and Sialon.

Concerning final density of cemented carbides after ERS with 7 kA, and 700 ms, Table 3 shows values measured by Archimedes method.

Table 3. Summary of ERS tests carried out with the different die materials and density reached in cemented carbides.

Material	**Number of dies**	**Number of ERS tests**	**Density (g/cm^3)**
AT/M	8	49	12.089 ± 0.275
NZP	14	36	12.023 ± 0.429
Z/M	3	4	11.912 ± 0.212
8YSZ	3	2	11.650 ± 0.212
Sialon	9	47	11.499 ± 0.328

As shown, sintered samples density is lower on using Sialon, although with differences almost disappearing when the scatter is considered for Z/M, 8YSZ, NZP and AT/M dies. Microstructurally, the bigger difference is observed with sialon dies, where a porous outer layer appears in sintered samples. Thus, new ERS experiments were carried out to check these results using more severe conditions (WC-6Co, 11 kA and 500 ms). The presence of the outer porous layer is clearly confirmed in the Sialon experiences when compared to any of the other options, as shown in Figure 3. For instance, the cemented carbides obtained using NZP dies are completely dense near the surface.



(a) (b)

Figure 3. Optical micrographs of the external surface of WC-6Co samples ERS with 11 kA and 500 ms by using (a) sialon and (b) NZP dies.

The lower thermal conductivity of NZP avoids superficial thermal loses, improving the densification of the material, whereas commercial Sialon dies lead to less dense metallic parts exhibiting a porous outer layer.

Also shown in Figure 3, a clear difference is observed on the ERS compacts surface roughness, with the Sialon die leading to a perfectly flat surface. Mean roughness (R_a) of the as prepared AT/M, NZP, Z/M and 8YSZ dies was between 0.5 - 1.5 µm (with peak individual values up to 2.5 µm). Commonly, measured R_a increased after every additional ERS experience. Roughness of the as received commercial Sialon dies was in the range 0.5 - 1 µm and remained almost the same after several tests, which is mainly attributed to the high dimensional tolerance and polished surface.

Results regarding dies surface roughness, measured after ERS experiments, are shown in Figure 4. Each point in the graph is the mean value from individual dies tested from each material. Cracked dies were not used for calculations. Values corresponding to R_a, R_y and R_z show a very similar trend.

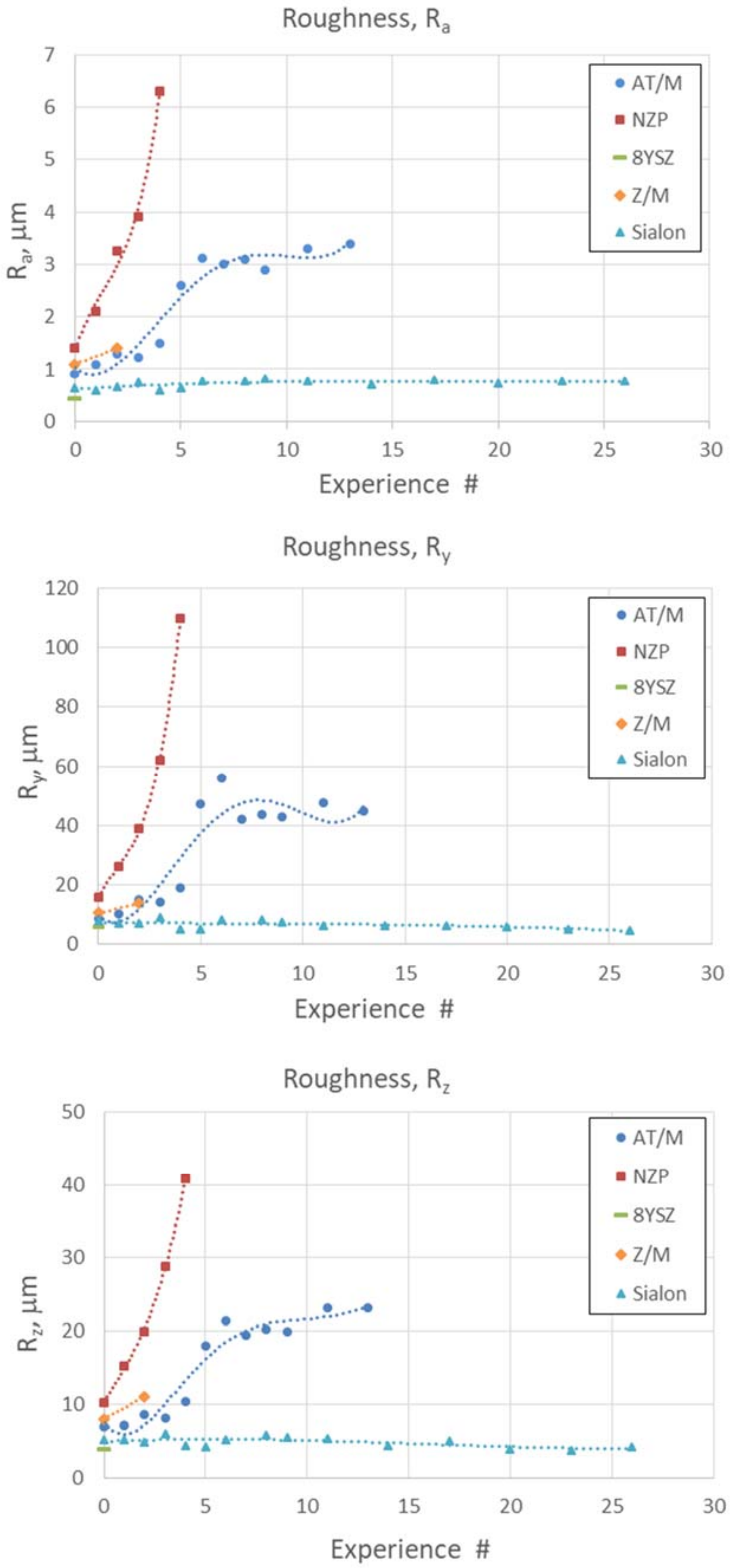


Figure 4. Roughness of the ceramic dies inner surface before and after ERS testing experiences.

In general terms, AT/M dies preserved the original roughness for about five tests, however roughness increased from 1 µm up to about 3 µm in the following five to ten tests. NZP showed a linear trend in the surface deterioration, therefore happening after a lower number of uses. The problem with 8YSZ, and also with Z/M, is the extremely low number of cycles supported by these dies, preventing any comparison with other materials. A very different behavior is observed with sialon, where roughness is not altered with the use of the die, remaining in a very low value.

In order to complete the study, the extraction force of the ERS compact was measured after each experiment (Figure 5). Regarding AT/M and NZP, and also Z/M and 8YSZ dies, the extraction force appears to increase after some ERS tests. On the other hand, sialon dies allowed the specimen extraction almost without effort. Measured extraction forces correspond to 12 mm in diameter punch pressures well below ERS cycle pressures. Nevertheless, the relative compact/die movement should be responsible of the observed quick surface deterioration. Consequently, dies lose tight geometrical/dimensional tolerances, nucleating cracks and failing.

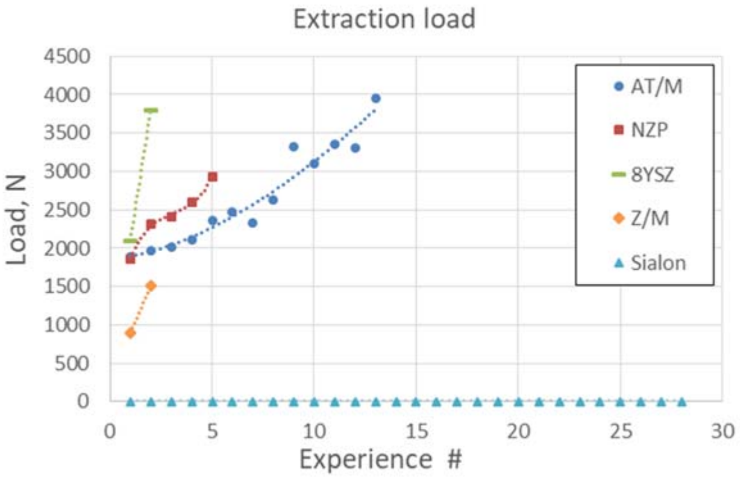


Figure 5. Extraction force of the ERSed cemented carbides from ceramic dies.

Another important observation about dies performance relates with the quality of the die wrapping procedure. Cracks were detected in the dies even after wrapping and before use, as shown in Figure 6a. The problem appeared when lead was cast in between a room-temperature set of steel and ceramic tubes. In this case, uneven heating of the inner ceramic tube (only the outer surface is heated by the molten lead) induces tensile stresses in the inner surface of the ceramic die, which usually cracks. That is why an alternative method of lead wrapping was used, namely, heating all the three materials together up to the lead melting point.

Figure 6. Cracks appearing in AT/M ceramic die a) during the wrapping process before ERS experiments, and b) after several processing cycles.

This surface deterioration can be the start for further damage on dies, leading to breakage after different cycles depending on the die material (Figure 6b). Nevertheless, as previously explained, other sources for dies cracking must be considered. A key point affecting dies performance and survival life relates to sintered parts extraction. Mechanical stresses pile up on dies walls as sintering progress, due to thermal expansion coefficients and stiffness differences between hard metals and ceramic die materials. Such stresses are withstood by the ceramic material as long as tensile stresses are well distributed over a certain length of the tube. However, when the part approaches dies mouth during extraction, uncompensated tensile stresses grow over ceramic die material strength. The problem can be overcome by transferring tensile stresses to the wrapping material. Nevertheless, when lead did not completely cover those end areas, no load transfer is possible and tensile cracks appear at dies mouth, as can be seen in Figure 6b.

Thus, Z/M and 8YSZ only withstood a maximum of 2 cycles; NZP, 4 cycles; AT/M, 13 cycles and Sialon lasting more than 25 cycles without breaking. It seems, according to the reported facts, that die durability problem could be tackled at least in two different ways. The first one consists in machining and polishing the dies surface. However, machining these ceramics materials is not an easy task, mainly when the dies thickness is relatively low. Additionally, in order to accommodate compact elastic expansion on die expulsion, a conical clearance should be prepared near the tube ends.

4. CONCLUSIONS

After the analysis of experiments results, conclusions can be summarized as follows:

- Several ceramic materials have been tested as dies during ERS experiments with WC-6Co powders. Tialite/mullite, zircon/mullite, zirconium phosphate based ceramic, yttria-stabilized zirconia and sialon, in general after conveniently modifying the original composition in order to achieve a low porosity, were studied. Porosities among 1.5 and 6% were achieved.

- The produced dies had to be reinforced by wrapping with a metallic rim. Shrink fitting process is of high importance as can induce cracking in the dies. Failure extension from mouth cracks occurs after every ERS process. To accommodate compact elastic expansion on die expulsion, a conical clearance should be prepared near the tube ends. Also machining and polishing should improve their behaviour. Initial roughness R_a of dies ranges from 0.5 to 1.5 µm. Dies roughness do not increase steadily till die failure. In several cases roughness did not increase at all before failure.

- 37 dies have been tested in this work, with about 140 samples processed. Experiences were carried out using 7 kA and current passing time of 700 ms. 8YSZ, Z/M, NZP, AT/M and sialon dies withstand progressively more ERS cycles before failure. Failure seems not to be related to the ERS action, but mainly to the extraction stresses, increasing as additional cycles are carried out except for sialon. Samples do freely came out of sialon die, with a very smooth surface expected for a commercial product.

- The lower thermal conductivity of NZP avoid superficial thermal loses, improving the densification of the cemented carbides, whereas commercial Sialon dies lead to less dense metallic parts exhibiting a porous outer layer.

Therefore, by improving the roughness of the NZP material with polishing and carefully wrapping the die with a metallic rim, NZP ceramic could be the best candidate material for effective ERS sintering.

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