Fabrication of bimaterial components by conventional powder metallurgy

A. Thomazic[a], Y. Le Guennec[b], Y. Kamdem[a], C. Pascal[a], J.M. Chaix[a], P. Doremus[a], D. Imbault[b], D. Bouvard[a], F. Doré[c]

[a] Laboratoire SIMAP, Grenoble University, BP 75, 38402 Saint-Martin-d'Hères, France
[b] Laboratoire 3S-R, Grenoble University, BP 53, 38041 Saint-Martin-d'Hères, France
[c] CETIM, BP 802, 42952 Saint-Etienne Cedex 9, France

Abstract

This paper presents the main results of a research programme devoted to the fabrication of bimaterial components by die pressing and pressureless sintering. Two materials with chemical compatibility and practical interest have been designed: a WC-Fe cemented carbide with high hardness and a Fe-W-C steel with high toughness. Two types of components have been fabricated, layered parts and tubular parts. Every step of the process (die filling, pressing, sintering) is described with emphasis on the main issues met during each one in relation with the component requirements. It is shown that the finite element simulation of pressing and sintering can help in optimising process parameters.

I. Introduction

Multifunctional components are made of different materials that have the required properties for various functions (mechanical, thermal, electrical, corrosion resistance, etc.). Nowadays, the assembly techniques used to obtain multi-materials involve long and expensive operations. In the future, powder metallurgy can allow cost-effective fabrication of components including several materials of different classes with a limited number of processing steps [1]. MULTIMAT research programme, which involved various French partners, has been devoted to the fabrication of bi-material components by conventional powder metallurgy, including die pressing and sintering. This paper focuses on the fabrication of components made of a tungsten carbide based material and an iron based material. Two geometries will be investigated: a layered one and a tubular one (Figure 1). In both case the requirements for the final component are the following: near net shape, strong and straight interface, uniform density distribution, no crack. The processing conditions will be described and the resulting components will be described. Emphasis will be put on the risk of crack formation. It will also be explained how the numerical simulation can help in optimising both die compaction and sintering steps.

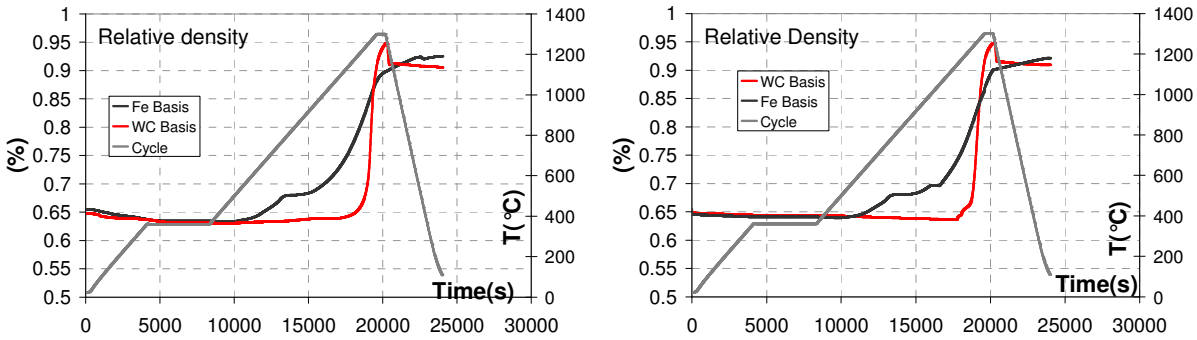
Figure 1: Schematics of the fabricated layered (a) and tubular (b) bimaterial components

II. Materials

The objective was to combine a tungsten carbide based material, with high stiffness and hardness, and an iron based material, with high toughness. To ensure chemical compatibility and to limit the number of chemical elements, the hard material (WC base material) consisted of tungsten carbide hard particles cemented by an iron base binder. The second material (Fe base material) was mainly composed of iron, with a small amount of tungsten

carbide and graphite powders. More specifically, we prepared two mixtures of Fe, WC and C (graphite) powders in the following proportions: Fe base (93.5 at.% Fe, 2 at.% W, 4.5 at.% C) and WC base (10.92 at.%. Fe, 44 at.% W, 45.08 at.% C). These compositions were chosen to optimise the sinterability and the interface development. Similar average WC and Fe particle sizes were used for the WC base powder mixture while for the Fe base one, finer WC particles have been chosen to improve carbide distribution in iron rich matrix and to reduce residual porosity after sintering [2]. The mixtures were processed through attritor-milling with 2wt.% polyethylene glycol (PEG 3400) dissolved in acetone. An organic binder was needed to promote compaction and to improve green strength in order to enable eventual green machining. Powder mixtures were dried and granulated through a 500 µm sieve.

Each mixture was uniaxially compacted into a cylindrical die (Ø=8 mm, H=6-8 mm) under 600 MPa. After compaction, the average values of green densities were 10.0 g/cm^3 (69 %) for WC base compacts and 6.0 g/cm^3 (74 %) for Fe base compacts. Before sintering, the compacts were held for one hour under He-4%H_2 flow at 360°C to remove the organic binder. After debinding, Fe base and WC base powder compacts were heated in argon atmosphere at 5°C/min and isothermally held at 1300°C during 60 minutes in a dilatometer with continuous measurement of axial length changes during heating. The resulting curves were accurately described in a previous paper [3]. The comparison of densification kinetics of Fe and WC base single materials shown in Figure 2a indicates that dimensional changes occur at different temperatures with different magnitudes. These differences could originate stresses and cracks at the interface of the two material components during sintering process. After cooling to room temperature, the experimental densities of the Fe base and WC base materials were 7.7 (96%) and 13.8 (94%) respectively.



a) b)

Figure 2: Densification kinetics of Fe base and WC base materials during sintering (1300°C - 60 min). Experiments (a) and modelling (b)

III. Modelling procedure

The numerical simulation will play an important role in the process optimisation. Modelling of die compression and of sintering of ceramic and metallic powders has been investigated in many publications [4-7]. Reliable experimental procedures and models have resulted from these works, and it is now possible to predict the behaviour of powders through the whole process of compaction and sintering using numerical simulation. However, very few studies concern the modelling of the processing of bi-material components.
In the present work the behaviour of each material during both pressing and sintering has been described by relevant constitutive equations. For pressing, a Ducker - Prager / Cap equation has been chosen, whereas for sintering we choose a classical equation that includes a free sintering densification term and Newtonian viscous term. Constitutive parameters describing pressing behaviour have been identified from three different tests:

diametral compression, uniaxial compression and instrumented die pressing test. Sintering constitutive parameters have been adjusted from data drawn from dilatometry experiences following the procedure proposed by Gillia and Bouvard [8]. Details of such experimental identification can be found in [9]. As an example, from the comparison of Figures 2a and 2b we concluded that the model predicts reasonably well the experimental data during free sintering.

ABAQUS finite element code has been used for the numerical simulation of each step of the processing of bimaterial components. Drücker Prager cap model is implemented in the ordinary version of the code whereas the sintering equations have been introduced with CREEP user subroutine.

IV. Layered components

Bilayer parts as the one drawn in Figure 1a have been fabricated. One of the mixtures (e.g. WC base) was poured first and slightly compacted in die (Ø=8 mm). Then Fe base mixture was added and pressed to 400 MPa to achieve a compact with 4 mm thick layers. Compaction parameters determine the density distribution inside the compact. For a two layer component formed by single-action pressing, density gradients depends on the order of die filling, i.e. whether the WC base layer is below or above the Fe base layer. If we assume that both materials reach full density after sintering, the two layers must have close relative density in the green compact so that they exhibit similar shrinkage during sintering. Numerical simulation was used to optimize the pressing conditions and search for the best distribution of relative density in each layer.

Figure 3 shows the relative density distribution calculated for both possible orders of the layers in a component with a mean density of 70%. The density when the WC base material is above the Fe base one is more homogeneous and is expected to give better results for sintering. It should also be noticed that the uniaxial compaction from the top of the sample induced a downward positive curvature of the interface between the two materials (Figure 3), which was also observed on real samples.

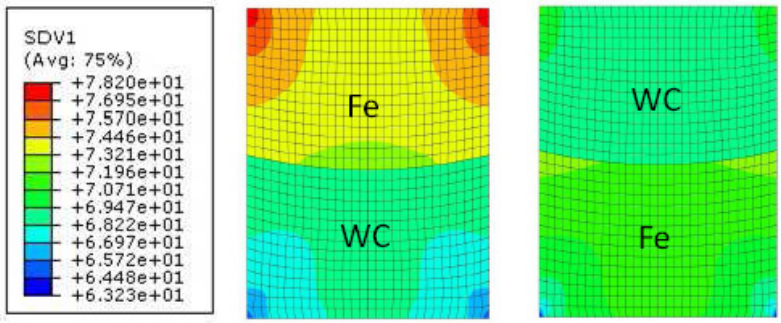


Figure 3: Numerical prediction of the distribution of relative density in bilayer components compacted at 570MPa

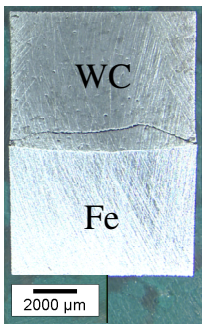


Figure 4: Fracture inside a bilayer part after sintering

The compact was next sintered at 1300°C during 60 min cycle. After cooling to room temperature, the experimental densities of the Fe base and WC base layers were 7.6 (95%) and 13.3 (91%) respectively. Several sintering parameters are expected to dictate the properties of the sintered bilayer material: graphite content in Fe base layer, co-compaction pressure, heating rate, sintering temperature and duration. A design of experiments (DOE) approach was used to analyse the effect of these parameters on sintering with a limited number of experiments [10]. This study highlighted the formation of liquid in the Fe base layer at 1300°C. Part of this liquid migrates towards the WC base layer by capillary

infiltration. This phenomenon has consequences on both materials: geometrical deformation, lower densification, new brittle phase formation (M_6C) at the interface between layers. The analysis of DOE combined to microstructural observations and numerical simulations indicated that a sintering at 1290°C during 30 min is a suitable cycle.

After sintering, some compacts exhibited cracks initiated at the sample surface at the two layers interface and propagated in the WC base layer (Figure 4). The question is: when did this crack arise: during compaction or during sintering? To search for possible cracks inside two layer green compacts, these compacts were axially cut and observed. No crack could be observed but the granular nature of green components could hide such cracks. Tensile tests were performed with bilayer compacts until breaking. Fracture always appeared in the material with the lowest density, whatever its chemical nature was, and never at the interface. These results suggest that cracks were not present at layer interface in the green samples, but appeared during sintering (or cooling). Experiments showed that having WC base on top position systematically leads to fracture despite a more uniform relative density and that using a curved upper punch might prevent cracking [11]. Internal stresses predicted by numerical simulation of sintering confirmed the experimental observations.

V. Tubular components

This component is formed of two coaxial rings, each one made of one mixture (Figure 1b). A critical fabrication step is die filling. The procedure that has mainly been used for this operation required the use of cylindrical concentric tubes made of steel to pre-compact the powders, as shown in Figure 5. During the first step, depending on which powder was introduced first, one of the tubes was set in the die and the filled powder (Powder A) was slightly precompacted in the second step. Then the tube was removed, hopefully without damaging Powder A. Powder B was next filled in the formed cavity and pre-compacted so that both powders had the same height. Note that another filling method involving an aspiration process and more suitable for industrial processing has also been investigated [12].

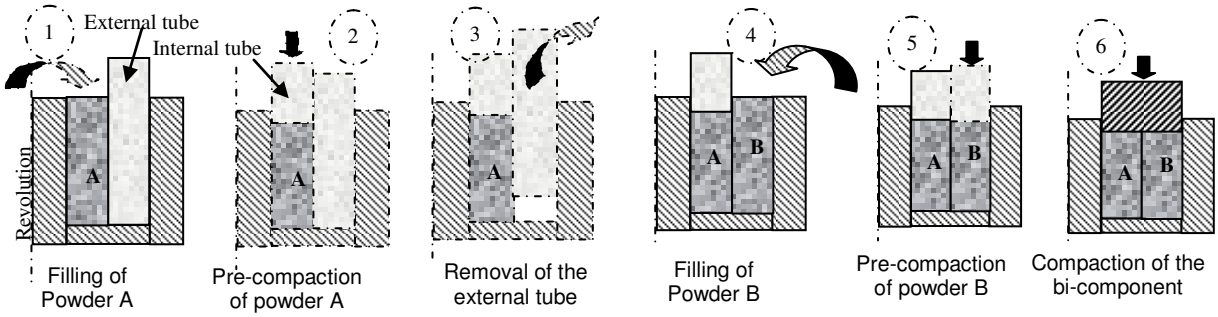


Figure 5: Schematics of filling and compaction process

Components with an external diameter of 27 mm and an internal diameter of 10 mm have been fabricated. The initial thickness of each layer was 5 mm for the internal ring and 3.75 mm for the external ring. Experimental tests were done for both possible configurations (Fe base in the internal ring and WC base in the external ring for the first configuration and the reverse for the second configuration). The mass of the powders was chosen so that we have an equivalent relative density in both rings after compaction. A pre-compaction stress of 42MPa was applied to the internal ring during the filling phase and the parts were pressed to 500 MPa. The final components (Figure 6a) exhibited enough strength to be extracted from the die and manipulated. However, when Fe base was in the external ring, thin cracks appeared on the external surface. In order to observe the shape of the interface between both materials, the components were embedded in an epoxy resin, cut and polished (Figure 6b). Note that the bright zones that can be seen along the edge of Fe base powder are due

to the infiltration of the resin. In both configurations, the shape of the interface was not straight.

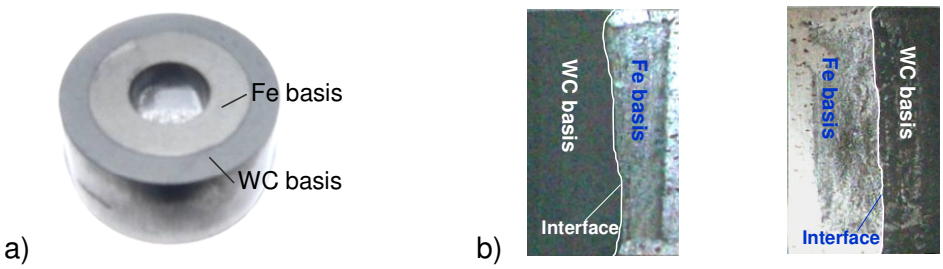


Figure 6: (a) A tubular component after compaction. (b) Cross section of tubular components after compaction.

The numerical simulation of the compaction of the bi-component has been achieved. Assuming a contact without friction between both powders gave the best results in terms of prediction of the interface geometry, as seen in Figure 7. This figure also shows the distribution of the density in the bi-component. In further studies, the numerical simulation of compaction will help us to obtain the ideal density distribution needed for the component after compaction to favour a safe sintering. The parameters that we will be able to change for this purpose are the kinematics for the punches, the height and the initial densities of the powders.

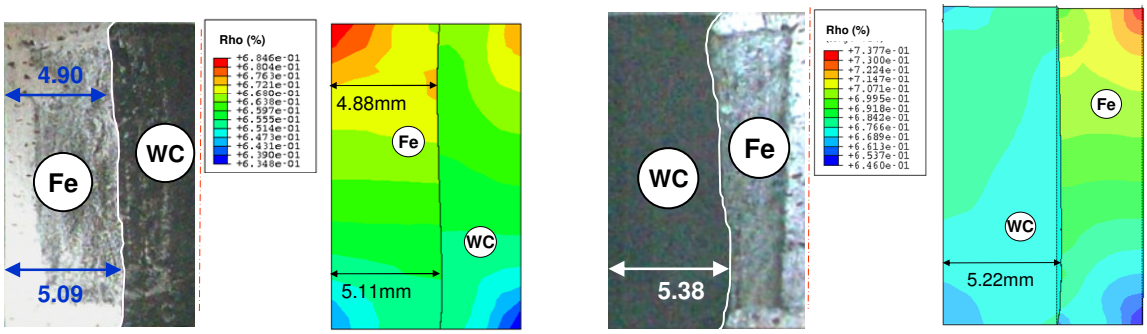


Figure 7 : Density distribution in tubular components predicted by numerical simulation

Green components were sintered in a tubular furnace at 1285°C for 1h in Ar atmosphere. Cracks appeared on the components after sintering in both configurations (Figure 8). The shape of the interfaces is the same as the one observed after compaction. A straight interface after compaction will probably lead to a similar interface after sintering. The interface was very weak in the bi-component with WC base in the external ring since it has been found to be broken after sintering. The cracks seemed to have initiated in WC base mixture (Figure 8b).

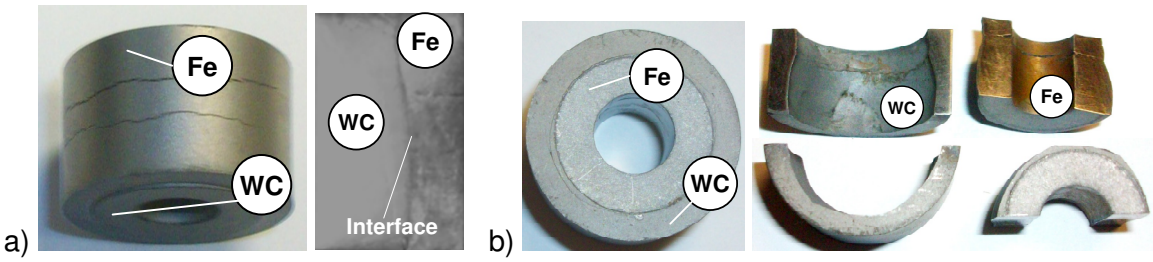


Figure 8: Tubular components after sintering. A) Fe base (external) - WC base (internal), b) WC base (external)-Fe base (internal)

The numerical simulation of the sintering of the components has been achieved. The predicted dimensions of the final components are close to the ones experimentally obtained. The simulation also provided the stress in the component at any time. Figure 9 presents the radial stress vs. time in either material nearby the interface for the case when the Fe base material is internal. The simulation predicts a strong increase of this stress around 500°C, when the sintering shrinkage of Fe base mixture starts. At that temperature, WC base mixture has no longer started to sinter and is still brittle. It is thus rational that this material breaks under tensile radial stress.

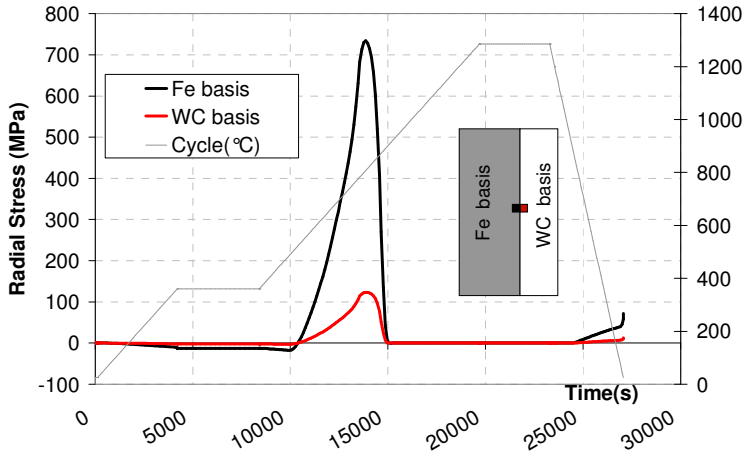


Figure 9: Numerical prediction of the radial stress close by the interface in both materials of a tubular compact with Fe base in the internal ring throughout sintering

VI. Conclusion

Cemented carbides and steel are compatible materials for co-sintering with a potential of application in hard/tough components. Bimaterial layered and tubular components made of WC-based and Fe-based powder mixtures have been fabricated by conventional powder metallurgy. Successful fabrication of such parts implies to control all processing steps: die filling, compaction and sintering. The association of experiments and finite element simulation has proved to be fruitful to understand the observed phenomena and to define guidelines for optimal powder metallurgy process of the studied biomaterial components.

Acknowledgments

The authors wish to thank Eurotungstene Poudres for proving the raw materials. They also acknowledge Direction Générale des Entreprises, Conseil Général de la Loire and Saint Etienne Métropole for their financial support of the *MULTIMAT* Project, within the Competitiveness Cluster ViaMéca and CETIM Foundation for its financial support of the "Multimaterial and Multifunctional Products" Project.

References

[1] R.M. German, D.F. Heaney, J.L. Johnson, Bi-material components using powder injection molding: densification, shape complexity, and performance attributes, *Advances in Powder Metallurgy and Particulate Materials*, MPIF, pp. 41-52, 2005.

[2] C. Pascal, J.M. Chaix, F. Doré, C.H. Allibert, Design of multimaterials processed by powder metallurgy: Processing of a (steel/cemented carbides) bilayer material, *Journal of Materials Processing Technology* **209** (2009) 1254-1261.

[3] A. Thomazic, C. Pascal, J.M. Chaix, Optimization of density, microstructure and interface region in a co-sintered (steel/cemented) carbide bi-layered material, *Ceramic Transactions* **209** (2010) *numéros de pages ?*
[4] H. Zipse, Finite-Element Simulation of the Die Pressing and Sintering of a Ceramic Component, *Journal of the European Ceramic Society* **17** (1997) 1707
[5] PM Modnet Research Group, Numerical simulation of powder compaction for two multilevel ferrous parts, including powder characterisation and experimental validation, *Powder Metallurgy* **45** (2002) 335
[6] H.G. Kim, O. Gillia, P. Doremus, D. Bouvard, Near net shape processing of a sintered alumina component: adjustment of pressing parameters through finite element simulation, *International Journal of Mechanical Science* **14** (2002) 2523-2539
[7] T. Kraft, H. Riedel, Numerical simulation of solid state sintering; model and application, *Journal of the European Ceramic Society* **24** (2004) 345
[8] O. Gillia, D. Bouvard, Phenomenological analysis of densification kinetics during sintering - Application to WC-Co mixture, *Materials Science and Engineering A* **A279** (2000) 185
[9] Y. Kamdem, D. Imbault, P. Doremus, D. Bouvard, Finite element simulation of the compaction and sintering of bi-material components, *Proceeding of EuroPM 2008 Conference*, Mannheim, EPMA, 2008
[10] A. Thomazic, C. Pascal, C., J.M. Chaix, Fabrication of cemented carbides/steel bilayered materials by powder metallurgy, *Proceedings of FGM'08*, Sendai, Japan, September 22-25, 2008. Référence complète ?
[11] A. Thomazic, Y. Le Guennec, Y. Kamdem, C. Pascal, J.M. Chaix, P. Doremus, D. Imbault, D. Bouvard, F. Doré, Influence of compaction on sintering of a two layer component, *Proceeding of EuroPM 2009 Conference*, Copenhagen, EPMA, 2009
[12] Y. Kamdem, P. Doremus, D. Bouvard, D. Imbault, Production of bi-material tubular structures by powder metallurgy, *Proceeding of EuroPM 2009 Conference,* Copenhagen, EPMA, 2009