Original Article: Investigating the Hot Extrusion Method for Cermet Powder Mixtures

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ABSTRACT

The process of hot extrusion of very fine cemented carbide powder with a combination of softener has been known for many years. This method has been successfully used for cermet's to make simple prismatic shapes that have a large length to cross-sectional area ratio. Cylindrical and quadrangular shapes and other sections can be easily made with this method, even making pipes is also possible. Depending on the plasticizer used (for example, polystyrene with the addition of dimethyl and diphenyl ether), the extrusion process requires temperatures between 160 and 175 degrees Celsius (320- and 350-degrees Fahrenheit). The process of complete and slow sticking under vacuum before full sintering is necessary and even vital to avoid stretching (from casting defects, from sagging), breaking or very small porosity. Twisting of bodies produced by extrusion similar to what happens in the plastic industry is acceptable for this process. In order to produce a high-quality product, hot pressing is necessary.

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Introduction

The process of hot extrusion of very fine cemented carbide powder with a combination of softener has been known for many years [1-3]. This method has been successfully used for cermet's to make simple prismatic shapes that have a large length to cross-sectional area ratio [4]. Another method of powder rolling is a common and well-known production process in powder metallurgy, which can also find applications in the production of cermet. In this process, cermet powder mixtures are guided from the hopper to the space between the rollers and removed as a compressed belt [5-7]. The way of placement of rolling rollers may be similar to conventional rolling on top of each other, in this case, the powder flows down from a funnel and through adjustable valves and through a channel [8]. The

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rollers are mainly placed horizontally. In this type of design, the powder enters the space between the rollers directly from the hopper. This type of feeding is known as "Saturated feeding" whose adjustment depends on the amount of powder located above the space between the rollers [9-11].

Compared to the beginning of the material turning into ingots between the rollers, the loose powder used in this process has no resistance before entering the rollers and must flow freely by itself or be forced into this gap [12-14]. During the process of compaction with rollers, the density and physical properties of the powder mixture change. For cermet powder compositions, the possibility of formability of a strip with sufficient density and strength depends on a number of factors, which of course are not limited to them, and these factors include the diameter and speed of the roller, the degree of loading of the cermet mixture with hard phase materials, the hammer ability of the phase metal and the amount of softener added to the mixture. The presence of hard components is necessary for the friction of the powders along the roller, as well as the internal friction of the powder mixture in the compression stage [15-17]. This is a desirable feature for cermet powders that are formed by rolling, which, of course, also reduces the strength of the cast piece in sheet or strip form [18].

The sheet thickness that can be pressed with a given rolling diameter is strictly limited. The ratio of rolling diameter to strip thickness is 600 to 1 and 100 to 1, which seems to be a range for various metal powders, and it is reasonable to assume that medium to smaller sizes are used for cermet’s [19-21]. Special devices are needed to prevent the powder from flowing out of the gap between the rolling rollers. An even flow of powders across the width of the strip is necessary to obtain the same density in the rolled strips. The edge may break. This is especially true for heavier bands. An optimal tape thickness must be obtained through experimentation [19]. Thicker strips are too hard to roll and thinner strips are too fragile. Rolling speed is another variable that can only be obtained during experimentation with a given cermet powder composition. Pure metal powders without hard phase are compressed at a rolling speed of 30 meters per hour (100 feet per hour) [20-22]. And this matter can be seen in every output close to this magnitude of speed that cermet’s are obtained.

A complete continuous powder rolling production line includes a unit of gluing and sintering furnaces, re-rolling stands and, if required, one or more annealing furnaces. Winding equipment is also required at the end of the production line. This equipment also includes capital-intensive molding materials created by guaranteeing demand for the product [23-25]. Although the labor costs for such an operation are small, this may be the case when this method of production was not yet found in the cermet’s. A simple composition is possible if, after the gluing stage (not shown) and the continuous sintering atmosphere, a product has enough strength and plasticity to be able to be turned into a coil [26]. The rolling compression arrangement was developed to produce a sandwich-shaped strip consisting of two strips of two different constructions.

Slip casting (suspension)

Slip casting is a method of shaping metal powders, a process that has long been used to produce ceramics. This method uses an aqueous solution of cermet powders (slip) that is poured into a plaster or porous plastic mold. Its liquid is absorbed by the mold [27], a tangent wall is formed with the surface of the mold, and in the case of hollow shapes, after reaching the appropriate thickness, the rest of the slip is removed from the mold, and then the formed part is separated from the inner wall of the mold;

For solid parts, leave the grout on until it dries slowly [28-30]. The slip intended for casting consists of a suspension of metal powder in water. The viscosity of the slip should be low so that it is easy to pour, it is stable all the time and the rate of settling or sedimentation is low. The piece prepared in this way should be easily separated from the mold and have low shrinkage during drying and high strength after drying. Mixing cermet powders can be a real problem, especially those powders that have a specific gravity difference between the hard phase and the binder metal [31]. This problem can lead to the creation of different composition and properties at the two ends of a cermet. The difference in the composition can cause the sample to crack during drying or sintering after that. In order to control the viscosity of the slip at an optimal level, it is usually necessary to use a particle cohesion inhibitor and also to control its PH. After slowly drying, the slip-cast part requires a bonding step by high-intensity sintering.

The resulting piece has a higher density than the density of the original powder mixture. Fine powders that are used intermittently to facilitate the slip casting process can lead to better properties in the sintered part. The advantages and disadvantages of slip casting are:

- ❖ Parts with large shapes and sizes can be made which are impossible to produce by pressing method.
- ❖ There is no need for expensive equipment [32].
- ❖ The best results are obtained from the smallest powders, which are also the most suitable for sintering.
- ❖ As a result, the final parts have excellent physical properties.

The main disadvantage of this method is that the process depends on the skill of the craftsman, and as a result, it is slow and expensive [33].

Suspension casting requires parameters such as slip viscosity and solution stability, wetting and dispersing agents, mold slip reaction and mold release. In the current state of technology, it is better to produce cermet parts with special complexity by injection into a metal mold than by slip casting [34]. The production process of the above-mentioned method requires more investment, but it works better for producing parts with medium to large volume.

Injection molding process (MIM)

This process has attracted many justifications since its first development in the 1970s. Commercialization of this method was slow. Mostly because the cycle required to deliver the parts accepted by the customer by sea transport was very long [35-37]. Much research and engineering applications of this method continued in many laboratories and grew much faster than expected.

In a laboratory, cermet parts are made with this process, and the commercialization of this method is on the way, especially in the field of cemented carbides. However, most of the current MIM testing is concentrated in the areas of ferrous and non-ferrous parts manufacturing. The process of injecting powder into the mold for cermet's consists of mixing and gluing metal components and hard phase powders together with a suitable polymer adhesive and then granulating the mixture [38-40].

The granulated product is heated and injected into the mold under pressure. The polymer properties added to the mixture reduce its viscosity to help form it, fill the mold and fill it evenly. After leaving the mold, the glue is removed and the rest of the cermet structure is condensed by sintering and perhaps by condensation and hot pressure. Adhesive composition and bonding methods differ between different MIM processes. There is no one-size-fits-all glue [41-43]. The first thing expected of the adhesive is to allow it to flow and fill the mold cavity.

It should also wet the powder and should be designed to minimize setting time and defects. A versatile adhesive that is not chemically soluble helps promote adhesive removal by:

Once one component is removed and the pores are relatively free, the remaining adhesive holds the particles within themselves and maintains the shape of the compressed part. Then the rest evaporates from the open pores without creating an internal vapor pressure that may cause the part to perish [44-46]. Waxes along with additives are the most used as adhesives, the phases of the molding operation are as follows:

- Tightening and filling the mold;
- Maintain pressure until the part solidifies;
- Return the filling mechanism;
- Opening the mold and removing the piece.

Mold filling depends on the viscosity of the flow from the main chamber to the mold chamber. Self-viscosity depends on temperature, shear rate, binder chemistry, powder interface chemistry, and type of loading. Thermal bonding is the most widely used method, but hair wicking and dissolution extraction can also be used as other methods [47]. Complete bonding is required before starting the sintering cycle. Most cermet's require a liquid phase sintering cycle to achieve full part densification. A modern furnace that combines the adhesive with the rest, sinters in a high vacuum, and has a final pressure sintering cycle that can do all these steps economically.

Applications and advantages of the MIM process for cermet's

In recent years, significant advances have been made in the use of the MIM process to produce components for heat engines, military hardware, computers and spacecraft, and machine components [48]. This has provided the opportunity to use the advantages of this method for the technology of cermet manufacturers. The general aspects of the application of the MIM process in the manufacture of Cermet are as follows:

- In principle, the MIM process is suitable for cermet's that do not require machining. Because the dimensional tolerances are very small.
- Production of complex shapes with small to medium sizes is possible with the MIM process. As long as the geometry of the shapes allows it to come out of the mold. Once this requirement is met, multiple surfaces, internal angles and cuts are acceptable.
- In small parts, the tolerance that can be achieved after sintering in conventional P/M parts with the MIM process is +3 and -3µm per mm (0.003 inch/inch). For cermet's, larger tolerances are required. will be to allow the cermet to shrink during sintering with the liquid phase for final densification.
- Small productions (as little as 2000 pieces) are feasible and practical for P/M departments. Due to the higher price level of cermet's and the relatively high price of cost-effective manufacturing methods, similar and even smaller size productions for cermet parts produced by the MIM process can be economically attractive.
- With proper gluing methods and sintering cycle with the liquid phase (which may continue with hot pressing), high quality parts with good physical properties can be produced with the MIM process [49].
- Additional coating of the mold occurs due to the hard phase during the injection of the compounds of a cermet into the mold, especially when the amount of loading is high, but in the MIM process, this issue does not seem to

be a serious problem. Future experiments and experiments will show whether there is a mold coating problem or not, and to what extent this affects the economics of using the MIM process to produce cermet's.

Defects seen in injection molding include inclusions and micro pores, which are caused by agglomeration and improper particle size distribution. But the bigger defects created in this method are divided into two categories: Defects caused by filling the mold itself, including incomplete filling, creating holes, and separation lines. Defects of solidification also include the germination of holes and creation of micro cracks due to residual stresses. Incomplete filling is a defect that can be easily recognized by observing the part and can be prevented by better temperature control and modification of the mold design. Porosity can also occur due to air entrapment during injection. But the separation lines are the areas where the injected materials are not properly welded together. These lines create disjointed or weak areas in the piece. These lines have a layered or folded appearance and remain as cracks after the baking process and greatly reduce the strength of the piece. One of the ways to solve this problem is to use the short shot (injection) method. This technique is such that the injection is stopped before the mold is completely filled, and by performing successive short intensity steps, a good picture of how the mold is filled for each type of geometry will be obtained.

Static hot compression

Hot compression is a cermet production method in which pressure and temperature are applied at the same time, the powder compounds are condensed both directly in the compression of the mold and in the form of cold pre-compression in the mold, and then to compact devices. The heat of the pressure sintering furnace is transferred. The pressures are significantly lower than the cold pressing method. These pressures can range from the dead weight of the device to 3MPa (500lb/in) for sintering pressure (for example in friction elements) or 10 to 35MPa (1500 to 5000lb/in) for hot pressing into the system. The liquid phase enters. Sintering temperatures are heated by resistance heating elements or by direct application of current through the mold by direct resistance or induction heating. In the previous case, the template material consisted of graphics. This is a more practical process because it usually does not require a device to control the atmosphere. The latter method requires ceramic molds that are sensitive to thermal shock, easily break when the product is ejected, and expensive to produce the exact dimensions of the opening mold. The advantage of direct heating of the part, during which the equipment and surrounding environment can remain cool, can be offset by thermal gradients and localized concentration effects in the product.

A controlled atmosphere is required for most systems with an oxidizing metal substrate. The densification effect in the conventional hot static densification method is more explained than the effect obtained by cold densification and subsequent sintering. The heating of the surface of the powder mixture, which occurs during the mechanical process, breaks the surface oxide layers, and produces bonding surfaces. Shape constraints are similar to those for cold static compaction. Single-surface prismatic parts with no internal cuts or angles are preferred, although shallow details on punched surfaces are acceptable. Large pieces work well with this method. A graphic tooled thermo-induction vacuum furnace is capable of both hot densification or re densification of 125mm (5 in) diameter ingots at pressures up to 90 mega grams (100tons) and temperatures as high as 2300degrees. Celsius (4200degrees of final phase) in complete vacuum with controlled atmosphere. The fully compressed part is removed while still hot to reduce cooling time, reduce the possibility of jamming in the mold, and extend the life of the mold. Items made in this type of furnace include WC-CO irradiated molds and friction elements, AL-B_4C ingots, stable oxide components, and some cermet's with a braid base.

Among the various processes of cermet production, static hot densification in the furnace of the same name is the only reliable single-step method to produce cermet with full density, high quality, and a piece close to the shape of a network of powder. However, the lifetime of the graphic mold is limited, and cermet compounds that do not react with the graphic are preferred. In fact, to prevent the cermet-graphic reaction, ceramic molds can be used for hot condensation. Although they are more fragile and more expensive than cermet molds. A complete hot press installation (including a vacuum system with an atmosphere generating power system, hydraulic system, controls and machinery) is expensive. In addition, the static hot press system requires a lot of effort. Because the products are only pressed one by one at a time. Therefore, hot pressing can be suitable for integration of sensitive and high temperature tasks for small to medium sized parts. For large and very large parts, vacuum hot pressing is often used. Because there are large equipment's available.

Hot Isostatic Press (HIP)

This method is becoming more popular as a means to produce carbide-based cermet's and other cermet's with very high and constant density. Internal defects and small and large porosity are eliminated as a result of production with this method. Equal pressure pressing is a continuous process that is carried out in a water-cooled pressure vessel that can withstand high internal pressure up to 210mega Pascal's (30 KSI). Increasing the temperature up to 1600 degrees Celsius (3000 degrees of the final phase) is done with resistance or high frequency furnaces that are installed inside the pressure vessels. The pressure transfer medium is a neutral gas, which is usually argon. Also, the medium of pressure transfer can be helium, which has a density close to that of water at the applied pressure (100 to 150mega Pascal's, or 15 to 20 KSI). More details and information about HIP equipment are available in Powder Metallurgy, Volume 7 of the 9th issue of the Metals Handbook.

Hot isostatic pressing was originally developed for use in high-pressure gas leak bonding processes, such as coating for nuclear fuel elements (for example, uranium oxide) with zirconium alloy coatings, and with applications such as powder consolidation and densification of agglomerated hard materials. The combination of cermet continued. Hot isostatic pressing is the most successful method used in the cemented carbide industry and among titanium carbide steel manufacturers. These cermet's, which do not easily tolerate high-density sintering, often have small local porosity up to 50µm; Because they have empty spaces in the range of 0.25 to 2.5mm (0.01 to 0.1inch), which is due to accidental or accidental pollution. Hot isostatic pressing is an improvement of the static hot pressing method, which has satisfied the requirements of this method for expensive and perishable molds. However, before the parts are heated for isostatic press delivery, they need a structure with sufficient density (even in the shell) to prevent gas leakage. Parts with lower density and internal pores need some gas pipe coating compound before any operation. There are three general methods for wrapping. In this method, the parts are formed and sintered up to 95% or more of the theoretical density, and according to the continuous results, the dense structure of the surface acts as an impermeable coating against the high gas pressure. On the other hand, the density rises to the required level until no internal porosity remains in the part. This method is mainly used for small and medium size parts.

The second method involves a can that is prepared to match the shape of the desired piece. A canister filled with a powder mixture that has been thoroughly compacted by shaking or pressing (a hydrostatically compacted or cold pressed part can be coated in a steel can instead of a powder mixture). After loading, the can is tightly sealed by welding and then unloaded. This method has often been used for medium and large sized parts and where the additional cost of a destructible can be ignored from an economic point of view. The third method is the "Uine-sejournet" process, where glass glaze is used to encapsulate the piece. This method can be more economical than the steel can method. All methods of preventing high-pressure gas infiltration are expensive. Fortunately, the HIP process is flexible enough to allow hot isostatic pressing of a number of freestanding or coated parts. The cost of the process can be divided according to the volume occupied by each piece in the available space of the furnace. Cemented carbide P/M products that have undergone the HIP process are hardly of higher quality than those produced by other processes. The higher quality is a result of the theoretical densities produced by the HIP process. Parts with a density close to the theoretical density have high surface resistance and more reliable physical properties. Hot pressing is an interrupted process that is extremely expensive; And when the cloaking method is used, it requires just as much effort.

Hot extrusion of cermet ingots

This method is unique among the different processes of Cermet. Because it is necessarily a solid state process. All compression processes described earlier involved a liquid phase. During hot extrusion of powdered materials, large hydrostatic compression forces occur. One of the components of the uniaxial force initially compresses the powder material to full density and then pushes the material into the mold. Depending on the characteristics of the front surface of the extrusion die, a shear component can absorb approximately half of the energy required for extrusion performance. The amount of final deformation of one step (the ratio of the cross-sectional area of the ingot to the cross-sectional area of the mold cavity) is greater than any of the other processes of cermet by hot method.

Conclusion

Combination of sintering and compression

The combination of gluing, sintering and pressing was done in a unified manner in furnaces, in an effort to simplify the manufacturing processes of cermet's and similar products. As previously explained, a sizing step prior to sintering of the fresh product containing lubricants, super glues or softeners is necessary. These additives must have different properties for static cold pressing, hot extrusion, powder rolling pressing, slip molding and cermet injection with slip molding. After gluing and during sintering with the liquid phase, the fresh products shrink to near full density. After the compression has progressed to the point where the pressurized gas could no longer penetrate the part, hot pressure compression occurs. The compressed cermet gas, which is now sintered, is compressed under pressure and temperature close to the formation of the liquid phase, which improves the quality of the product by eliminating the porosity of the part, internal cracks, and its defects.

Recent tests with WC-Co parts have shown that using lower isostatic pressures as low as 2.7MPa can produce parts with strength and density nearly equal to parts produced by the high-pressure HIP process. Based on these findings, pressure furnaces work at a temperature between 1450 and 2200 degrees Celsius (2640 and 3390degrees of the final phase). This new furnace, which performs a combination of three operations in one cycle, offers the benefits of sintering and hot compression densification as follows:

- Gluing, sintering and densification are done under pressure in one cycle and in one container.
- During the different stages of the processes, the parts are not exposed to air.
- Controlling the entire cycle with an electronic microchip, it has automatic operation and also has a high degree of production programs.
- The transfer of parts from one process stage to another is prevented, which in turn reduces the work cost and process time.
- Centralizing processes saves energy.

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