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Overview of conventional technologies using the powders of metals, their alloys and ceramics in Industry 4.0 stage

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ABSTRACT

Purpose: Among the technologies that play a crucial role in the current stage of development of Industry 4.0 conventional powder engineering technologies are of great importance. Based on a comprehensive literature review, conventional technologies using the powders of metals, their alloys and ceramics are described. Development perspectives of the most widespread among them were indicated.

Design/methodology/approach: Extensive literature studies on conventional powder engineering technologies have been carried out. By using knowledge engineering methods, development perspectives of individual technologies were indicated.

Findings: In addition to the presentation of conventional sintering technological methods, sintering mechanisms in solid-state and liquid phase sintering which accounts for 90% of the commercial value of sintered products are presented.

Originality/value: According to augmented holistic Industry 4.0 model, many materials processing technologies and among them conventional powder engineering technologies play a key role in current industry development. For this reason, these technologies have been characterized in detail on the basis of available literature sources.

Keywords: Powder engineering, Conventional powders manufacturing methods, Powder metallurgy, Liquid phase and in solid-state sintering, Manufacturing of powder products, Holistic augmented Industry 4.0 model

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MANUFACTURING AND PROCESSING

1. Introduction

The current stage of Industry 4.0 is the beginning of a new era of digital industrial technology, and the corresponding Industry 4.0 model introduced in the original reports [1-4] proved to be incomplete. In fact, it concerns cyber-information systems, and the criticism of this incomplete approach has resulted in the development of the authors' extended holistic model Industry 4.0 [5-8]. The most important is that in the technological plane of this extended holistic model, one of the four components are technological processes and not only incremental methods that were indicated only in the original model. Among these technologies, the importance of technologies using the powders of metals, their alloys and ceramics are important. In [9], an overview of all the most important technologies that are relevant in this context is given. The paper [9] describes the methods for manufacturing powders of metals, their alloys and ceramics, and the following papers [10,11] describe conventional and additive technologies for products manufacturing using these powders. This paper includes selected from among them, listed in Table 1.

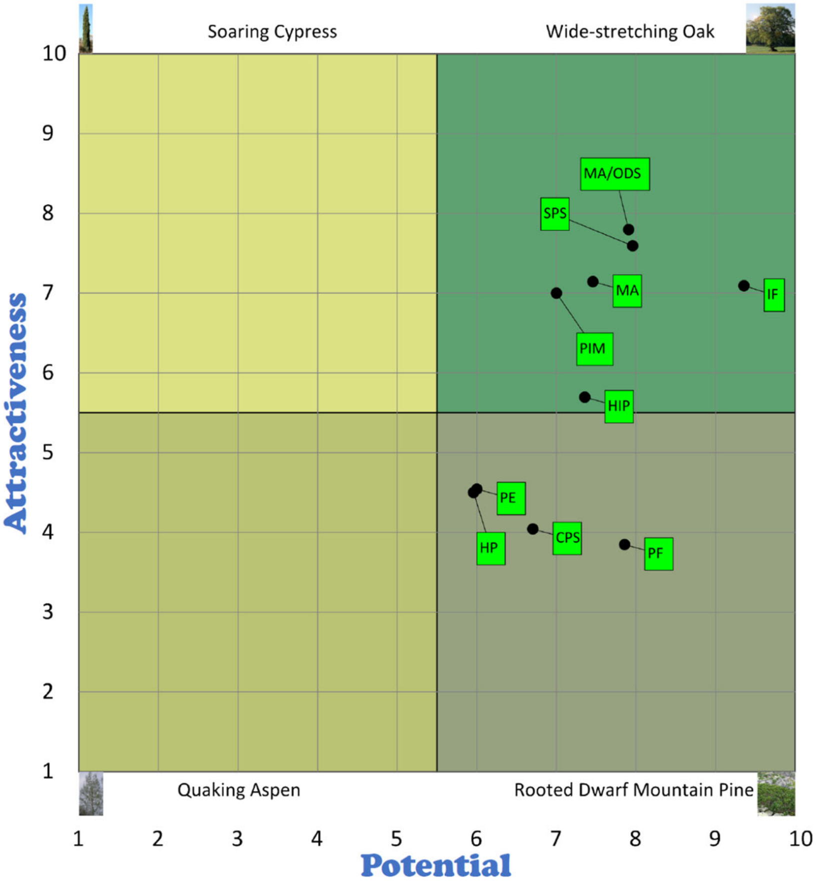

Fig. 1. The attractiveness matrix of technologies taking into account conventional and additive technologies using the powders of metals, their alloys and ceramics

Table 1.
List of conventional technologies in which the powders of metals, their alloys and ceramics are used to manufacture final products

Powder metallurgy technologies		
1	CPS	Conventional pressing and sintering
2	HIP	Hot isostatic pressing
3	HP	Hot pressing
4	IF	Infiltration
5	MA	Mechanical alloing
6	MA/ODS	Mechanical alloing & Oxides Dispersion-Strenghtened
7	PE	Powder extrusion
8	PF	Powder forming
9	PIM	Powder injection mouduling
10	SPS	Spark plasma sintering

In paper [9] the procedural benchmarking method is used involving the comparative analysis of these technologies in terms of their potential and attractiveness. [12-19] conventional powder engineering technologies were indicated among the considered technologies that can ensure business success. Hence, among the detailed criteria were the costs of initial investment related to the purchase and commissioning of the machine park, as well as the costs of the production process, product quality, meaning precision of production, production efficiency, as well as environmentally friendly, including energy consumption, use of recycled materials, production safety, determining the potential of technology. On the other hand, the criteria for assessing the attractiveness of technology include the range of applications in various industries and versatility, technological advancement reflecting the technology life cycle stage, investment payback time, development perspectives that can be translated into profits in the enterprise and scientific research realization.

Using the authors' computer software, the numerical results (from 1 to 10) were calculated and Figure 1 presents the dendrological matrix referring to the technologies selected in this article. Technologies with high potential and attractiveness are located in the "Spread oak" quarter when the less attractive ones are located in the other quarters of this matrix. Technologies located in the Quivering Aspen quarter are the least promising. Mechanical alloying and its variant enriched with Oxides Dispersion-Strengthened and spark plasma sintering took a high position in the conventional powder engineering technologies group. The widely used powder injecting moulding and hot isostatic pressing also has a strong position, and the infiltration position is promising.

The purpose of this paper is to present conventional technologies using the powders of metals, their alloys and ceramics based on a comprehensive literature review. Detailed knowledge of these technologies is of particular importance due to the need for their proper automation, computerization and digitization required by the Industry 4.0 approach rules.

2. General classification of powder metallurgy as the basic technology for the manufacturing products from the powders of metals and their alloys and ceramics

Powder metallurgy is a technical field involving the manufacturing of semi-finished and products from the powders of metals and their alloys and ceramic materials without having to melt the main component [20-27]. Classical powder metallurgy, however, is based on pressing and forming the powder in such a way that after sintering the product has the dimensions and shape of the finished product or semi-finished product, requiring only a small final treatment. The history of powder metallurgy dates back to 3000 BC since elements in this technology were already manufactured in ancient Egypt. The ancient Incas made a precious stone from gold and other precious metals. The start of mass-production dates back to the end of the 19th century. Initially, fine particles were extracted from the sponge iron, which was then melted down or sintered. It wasn't until later that metal powders began to be sintered. Currently, powder metallurgy plays an essential role in material processes technologies and product creation and engineering materials properties. Thanks to powder metallurgy processes, it became possible to reduce employment, significant production capacity was freed, it became possible to reduce the use of materials and advantageously reduce waste. Powder metallurgy can be competitive with other manufacturing processes, such as casting, plastic forming and machining, by supplementing or replacing these technologies. The justification is usually a large production scale of 1000-10000 pieces, enabling profitability of return on investment in technological equipment and production accessories. It is estimated that the automotive industry is currently the primary user of moulded and sintered components using metal powders. At present, approximately 80% of the parts made by powder metallurgy are used in the automotive industry. Powdered and sintered gears for gearboxes are characterized by hushed operation, which is associated with the damping of vibrations in porous materials. The teeth of such gears are polished and heat-treated, which ensures their high hardness and wear resistance. Uniaxial stamping in closed dies is still the most common and is dedicated to symmetrical elements with relatively simple shapes. In addition, powder metallurgy methods are used in other industries, including in the aviation, energy and household devices industries, and increasingly in medicine, implantology and dental prosthetics. Powder metallurgy methods produce components with a complex shape and narrow dimensional tolerances, often with relatively small sizes and on a mass scale. The technological process of powder metallurgy provides high flexibility in the selection of physicochemical properties and density while ensuring the required precision and upper surface quality. Powder metallurgy methods have found application in the mass production of many components made of iron alloys, including steel, alloys of other metals, but also cemented carbides and other sintered materials including sintered tool materials. Sintered machine components are used in the machine and automotive industries. Gears, rollers, washers, nuts, pawls, shock absorber parts, valve seats, bearings, building joinery parts, reinforcement parts, office machines parts and sewing machines parts are made of sintered powders.

Advantages of this powder metallurgy technology include minimizing the number of technological operations relative to machining or plastic forming and a lower processing temperature than conventional metallurgy, the possibility of maximizing the consumption of input materials in at least 95% of cases and minimizing the specific energy consumption related to manufacturing 1 kg of ready-made elements compared to other technologies. Powder metallurgy, however, promotes obtaining materials with relatively high porosity, and thus a relative reduction in strength, although this may have a beneficial effect in some technically justified cases. The disadvantages of powder metallurgy methods include the difficulty of obtaining products of a complex shape and a relatively large mass due to the uneven distribution of pressure in the powder volume during pressing. Powder metallurgy, however, is unrivalled when manufacturing about 43% of materials and products.

This applies in particular to cases where the expected property is the porosity of the product, e.g. in special filters and in self-lubricating bearings. The porosity of the sintered metal skeleton can be used to infiltrate other liquid metal with a lower melting point, as in electrical contacts or some parts for medical application for example as cells scaffolds. Some components can only be manufactured by powder metallurgy, e.g. some high-melting metals and cemented carbides.

In general, in the classical technological process of products manufactured by powder metallurgy, in addition to the production of metal powder or mixtures of various metals, the following operations can be listed:

- powder preparation and mixing with lubricants and sliding components,
- cold forming and compaction,
- sintering,
- finishing.

Figure 2 shows [21] a general classification of sintering methods for materials used in powder metallurgy. Figure 3, on the other hand, contains a sequence of technological operations specific to conventional powder metallurgy [21], with examples of several methods for producing powders. Figure 4 shows, for example, the classic method of manufacturing cemented carbides as one of the mass products manufactured by powder metallurgy technologies that cannot be manufactured by other technologies.

Sintering powders in the form of cold-formed preforms or loosely connected powder particles consists in their joining under the influence of heat, as a result of which metal sinters and sometimes composites are produced, including ceramic-metallic ones called cermets with specific mechanical and physicochemical properties [20,21, 28-31] (Fig. 5). Sintering can be carried out freely or under force, i.e. in combination with forming to provide a certain shape, e.g. by hot pressing powders, hot rolling or hot forging. The sintered material is always somewhat porous and has a single or multiphase structure. The individual constituent powders are combined in sintered materials, new grains are formed in the sintered material, the sinters differ from the input powders by their properties, volume and density [20,21]. Sintering is an irreversible process, so it is impossible to repair errors caused during mixing and forming powders, with many damages being more noticeable after sintering and are often, though incorrectly, interpreted as sintering errors [20].

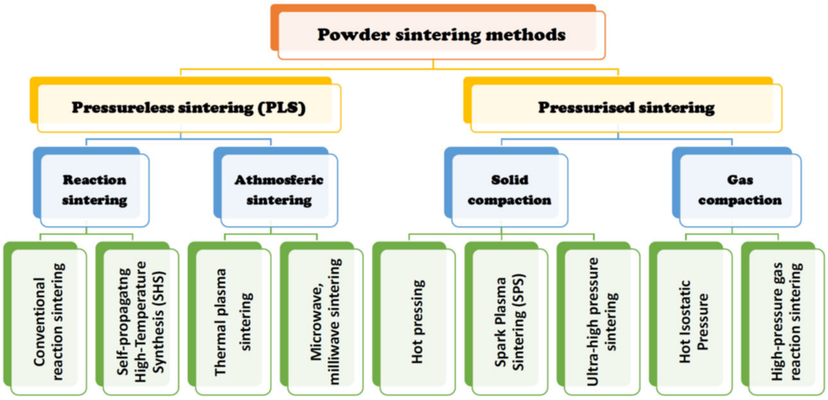

Fig. 2. The general classification of sintering methods for materials used in powder metallurgy

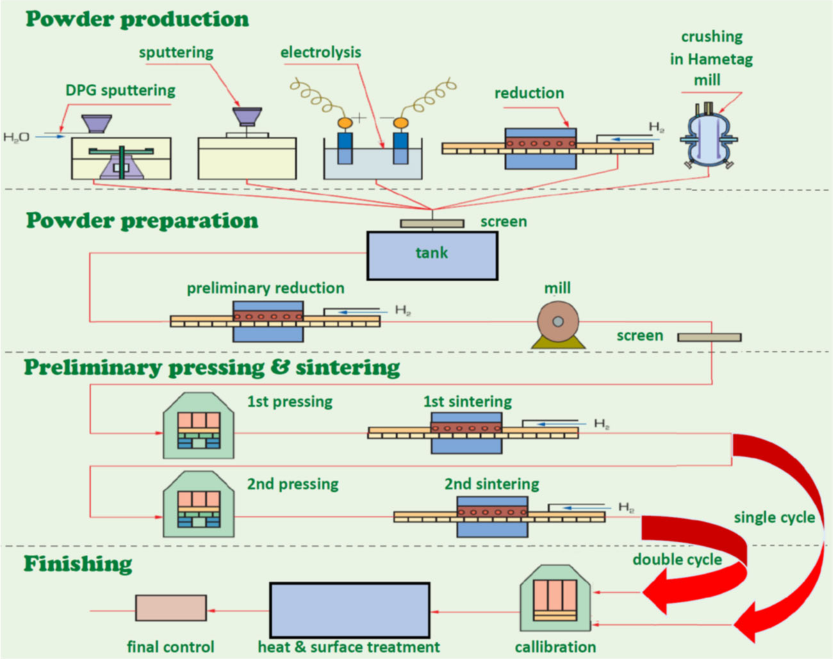

Fig. 3. A simplified diagram of the mass manufacturing of iron and steel powder products

In the final phase, materials produced by powder metallurgy are subjected to sinter finishes involving various, generally alternative operations. Cold or hot pressing again, including hot isostatic pressing, ensures the improvement of mechanical and physical properties of metal sinters. In order to achieve high dimensional accuracy, calibration is used, with loads being much lower than during cold forming. Ensuring the assumed geometrical features and improving the properties of semi-products made of sintered metal in the shape of blocks is made possible by plastic working, e.g. forging or rolling. Heat treatment is often used, depending on the chemical composition, including quenching and tempering, supersaturation and ageing, surface quenching [20,21,32-34]. The final shape and the required surface smoothness is ensured by precision machining, mainly grinding. A thermo-chemical treatment can also be applied, including carburizing, carbonitriding or nitriding and passivation [15]. Coatings can also be applied by physical and chemical vapour deposition methods PVD/CVD and atomic layers deposition ALD, including to increase hardness, corrosion resistance as well as barrier properties and aesthetics. Surface plating and electroplating are also used to improve corrosion resistance or for decorative purposes. The burrs are removed and burnishing is also used as the plastic deformation of a surface due to sliding contact with another object [15,21,32-34].

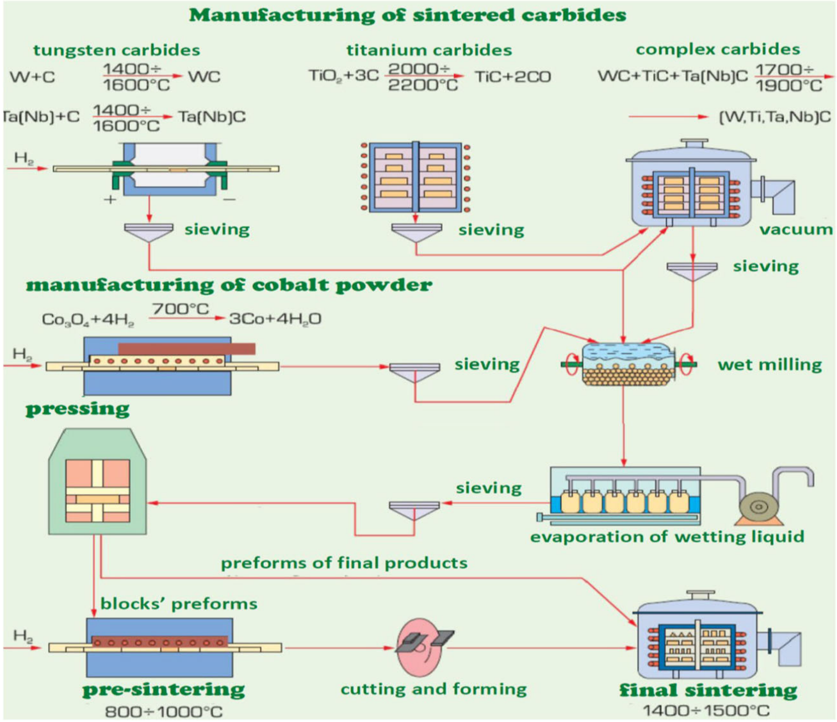

Fig. 4. Diagram of a classic cemented carbides manufacturing technological process

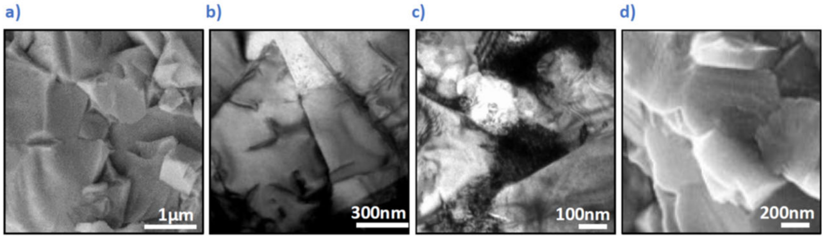

Fig. 5. Structure of a) cemented carbides breakthrough (SEM); b) WC carbide in the structure of cemented carbides (TEM, thin foil); c) TiC carbide in the Coβ matrix (TEM, thin foil); d) cermet breakthrough (SEM)

3. Phenomena occurring during preforming and subsequent sintering the powders of metals and their alloys and ceramics

The essence of powder metallurgy is determined by phenomena occurring during preforming and subsequent sintering. Cold powders can be compacted only to the limit value of space-filling, due to the limited deformation possibilities of individual powder particles, regardless of the fact that only the particles of raw materials used in ceramics are rigid, and the particles of metal raw material can undergo plastic deformation. According to the percolation theory, the powder filling factor at room temperature is approx. 0.6. Cold powder forming consists in compression in a closed space causing its compression, in a way depending on the shape of the matrix and the properties of the powder, especially its plasticity and formability [21,23] given below:

- cold pressing in closed dies;
- isostatic pressing in high-pressure chambers;
- vibrating compaction of powders;
- circumferential pressing;
- rolling;
- powders' rolling
- cold extrusion of powders;
- impact forming;
- cold forging;
- the casting of slurry (i.e. suspension of the base material powder in a liquid with the addition of agents to prevent agglomeration of particles).

In order to further compact the space-filling and consolidation, thermal activation is necessary. Due to the increase in temperature, atomic (ionic) vibrations are activated, which on the surface of the particles increases the number of bonds and the formation of permanent bridges between the particles. Their surface increases, limiting the possibility of relative movement of particles but activates mass transport within the particles. Eventually, the shape of the particles changes and the pore volume systematically decreases.

Solid-phase sintering takes place at a temperature below the melting point of the main component. Forming causes adhesion of the powder particles in the solid phase and as the temperature increases, the diffusion of atoms initially on the surface and overtime in the entire volume of the powder.

Powder particles combine with each other, which results in a successive decrease in sinter porosity. The driving force for solid-phase sintering is the surface energy of particles, which tends to be minimized, which reduces the area of free surfaces. Bridges form between the individual particles, their surfaces are smoothed, after which the particles undergo spheroidization and the pores disappear. Since sintering occurs in the solid phase (Fig. 6), the dominant mechanisms of matter transport are particles boundary slide and diffusion as well as evaporation and condensation. In the case of sintering with forming, under the influence of applied forces, plastic deformation of the metal powder particles occurs, and depending on the temperature range to which the powder is heated, recovery and recrystallization occur, respectively either static or dynamic.

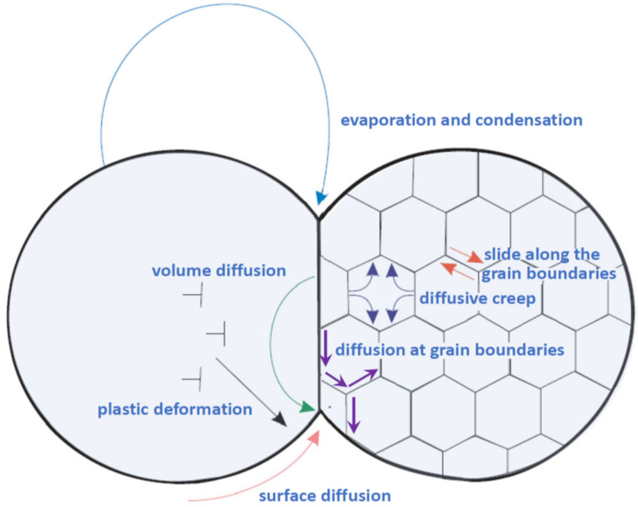

Fig. 6. Diagram of matter transport mechanisms in solid-state during sintering

Liquid phase sintering (LPS) is a consolidation technique of powder compacts containing more than one component at a temperature above the solidus of the components and hence, in the presence of a liquid. LPS occurs at a temperature above the melting point of the lowest fusible of at least two powders forming the mixture. Sintering in the liquid phase accounts for 90% of the commercial value of sintered products [35]. Liquid phase sintering (LPS) is defined as sintering involving a coexisting liquid and solid during part or all of the sintering process [36]. The liquid phase formed under these conditions should be able to wet the particles of the remaining insoluble powders contained in the mixture [37,38]. The occurrence of the liquid phase between molecules induces capillary forces [39,40], which cause densification and, as a result, the melting of the solid in distant areas with high surface energy, and their subsequent solidification [36]. Liquid phase transport is hundreds of times faster than possible in the case of sintering in solid state [35].

Sintering with a liquid phase usually occurs when using multi-component powders, especially those that form low melting eutectics. Alloy additions with high surface activity as well as an increase in system temperature are conducive to changing surface energy and improving the wettability of the liquid phase, which increases sintering intensity [41]. Sintering activators are often added to sintered material powders in the form of boron, copper, phosphor copper, molybdenum carbon, tantalum, titanium, vanadium, tungsten, and the process is called activated sintering [22,42]. Of course, the choice of these sintering activators depends on the type and chemical composition of the primary sintering powder. A properly formed liquid phase causes a fast grain connection, but also promotes microstructural coarsening. Most often, the particles are mixed to start the liquid phase formation process [35]. A liquid forms between the powder particles, filling the pores between them. Small particles can be completely melted, while large ones usually remain partially melted on the surface, as a result of which they rearrange and solidify new particles from the liquid. Most often, the liquid is kept in the high-temperature part of the process, although other types of sintering with liquid phase may also occur. During permanent sintering in the liquid phase, the liquid is present during sintering [43]. Transient liquids and partially transient liquid phase sintering systems arise from the melting of additive powders or their reaction with each other or with base powders [36]. Liquids resulting from the melting of additives and base powders should have complete solubility in the liquid state without any phase separation to ensure unrestricted mass transport and liquid spreading during sintering [44]. The low solubility of the additive in the solid base powder is required. It results in its segregation to the particle boundaries. It promotes an increase in the proportion of liquid, which in turn causes contraction associated with re-coagulation or, on the contrary, swelling if the solubility of the additive in the base powder is high (Fig. 7) [36-38]. In transient sintering with a liquid phase, liquid only occurs in the early stages of sintering [43]. In transient sintering, the additive is completely soluble at the sintering temperature in the solid powder base, and the liquid content is completely reduced during sintering [41]. As a result of the exothermic reaction of various powders, reactive sintering with a liquid phase occurs. It is still possible to infiltration sintering and activated liquid phases sintering [35]. Subsequent progressing sintering stages involving the liquid phase include liquid spreading and fragmenting the solid bonds, grain rearrangement, solution's resolidification, and finally, solid skeleton sintering [35]. It leads to the creation of a new compact arrangement of particles that adhere to each other and are sintered. Particles are rearranged in the presence of a high wettability liquid phase that fills the space between the solid particles, resulting in a radical reduction in porosity and shrinkage of the sintered product or component [45-49]. The structure of sintered materials with the participation of the liquid phase consists of uniformly distributed solid phase particles in the matrix of solidified liquid.

An illustration of the sequence of liquid phase sintering mechanisms includes successive particle rearrangement to allow for compaction, contact flattening of the particles together with re-solidification combined with the reduction of the distance between particle centres and coalescence of solid particles associated with solid-state sintering [50]. This approach is greatly simplified because it has not been properly proved experimentally [39], the relative movement of individual particles [51] as well as grain growth by re-coagulation [52] have not been taken into account with the criticized assumption that coalescence of solid particles is following without liquid between particles powder [36].

Liquid-phase sintering (LPS) not only used during sintering of metal powders and their alloys, but also during sintering of ceramic materials, including Al_2O_3, ZrO_2, TiO_2, $Pb(Zr,Ti)O_3$, ZnO, mullite, SiC, Si_3N_4, and SiAlON, for structural, electronic, chemical, and thermal applications [53,54]. However, in the case of advanced technical ceramics, the volume of the liquid phase is generally limited to no more than 5% by volume [55]. The volume of liquid is insufficient to fill the inter-particle pores, which has implications for the compaction mechanism.

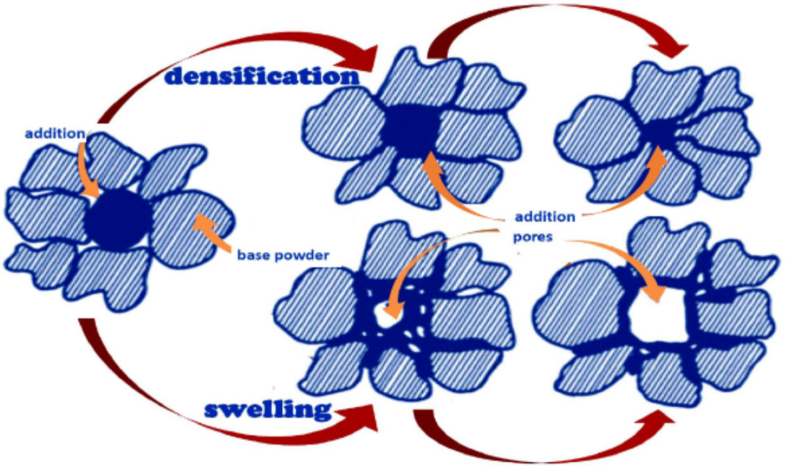

Fig. 7. Diagram of structural changes during sintering in supersolidus conditions

Sintering supersolidus is a one of type of the liquid-phase sintering. It occurs in the case of high-carbon steels, high-speed steels and nickel-based superalloys (Fig. 8). The particle temperature is kept above the solidus temperature. Then, the finest particles are melted, and large particles are partially melted [48,56-59]. Shrinkage of the preform is particularly significant in these cases. It is also the other way round and the sinters swell when using mixtures of two-component powders A and B, with the diffusion rate of component A to B many times greater than component B to A. It is accompanied by high diffusion porosity, which can be used to make porous preforms.

4. The sintering of metals and their alloys powders by extrusion, pressing and forging

Conventional powder metallurgy methods have gained many refinements and modifications that can be considered as special powder metallurgy methods.

Powder extrusion (PE) is made with a binder or plasticizer cold at room temperature with addition, and hot without reinforcement. During the cold extrusion, the metal powder is pressed in the matrix, as a result of which there is extrusion through the hole in the base of the matrix (Fig. 9a).

Powder cold extrusion requires up to 50% binder content, including paraffin wax, starch, benzol, resin or shellac. The binder is removed during sintering of the cold-extruded material, resulting in significant porosity. This process is used for the manufacturing of cemented carbide drills and

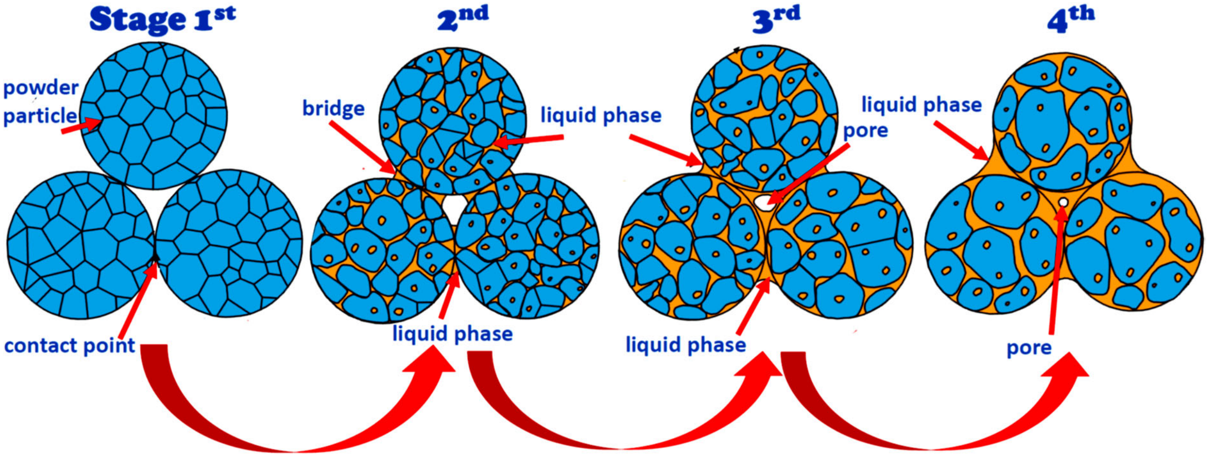

Fig. 8. Diagram of subsequent sintering stages of supersolidus

milling cutters. In the case of hot extrusion, the powder mixture is pre-concentrated in the can by thermal compression until all air has been removed. Then the lid is tightly welded to can. It prevents the oxidation of powders at high temperatures up to 1400°C for some refractory metals. The sealed can of mild steel is then tamped with a stamp until it becomes die-shaped (Fig. 9b). Further pressure causes long sections of material to be extruded through the hole in the matrix base. High extrusion rates can be obtained, which leads to good powder density almost to theoretical density. The process is used for many materials, including cemented carbides, SAP sintered aluminium powder, refractory metals, including tungsten and molybdenum, beryllium, cermet elements, magnesium alloys, nickel-based alloys. This process is sometimes used for the manufacturing of pipes and profiles of various metals and alloys with a diameter of several to several dozen mm and a length of 3-30 m. The PE process is commonly used for the production of tool cermets as well as wires and spiral drills with a diameter of up to 0.5 mm from cemented carbides. In their own research, the authors of this paper used this technology to produce bars from composite materials with a matrix of aluminium alloys reinforced with halloysite and carbon nanotubes [60-62].

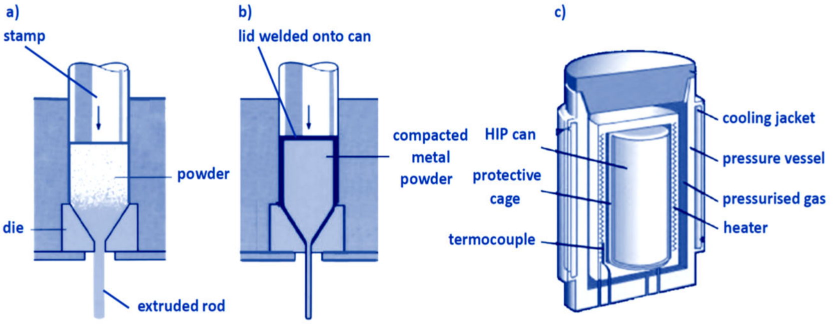

Fig. 9. Diagram of selected powder metallurgy processes; a) cold pressing; b) hot pressing with can for pre-pressing and degassing of the powder; c) hot isostatic pressing

Hot pressing (HP) involves placing loosely filled or pre-compressed powder in a graphite or steel mould to heat to 2400°C for steel, and subsequent pressing at a pressure of up to 50 MPa exerted on the punch in one or two cylinders. In the case of induction heating, the mould is placed inside the induction coil generating a high-frequency electromagnetic field or in the event of indirect resistance heating in a chamber electrically heated by graphite elements and transmitting heat by convection. Under these conditions, sintering and creep occur as a result of the synergic interaction of thermal energy and high pressure with a small proportion of deformation [32,63,64]. Re-pressing occurs as a result of grain boundary migration and plastic flow of material in their area after sintering. In the case of induction heating, sintering can occur with a liquid phase, even at low pressure. In the case of resistance heating, a high processing temperature can be achieved. However, this generally requires a long heating time, which is a significant drawback of this method. This method is mainly used to produce hard and brittle materials, including diamond composites for tools and for technical ceramics [20,65].

Hot isostatic pressing (HIP) takes place in a high-pressure chamber (Fig. 9c) at an elevated temperature, about 0.8 solidus temperature and under isostatic pressure of a gas, usually argon. Other gases or gas mixtures are used in special applications. The chamber is heated, hence the pressure in the tank increases, and most often, the device is equipped with a gas pumping system providing a pressure of 50-300 MPa [66]. Internal voids and microporosity are simultaneously eliminated, and the density is increased by a combination of plastic deformation, creep and diffusion welding. As a result of that, consolidation and re-pressing of sinters occurs, in particular with the help of a metallic matrix [32,64, 66-68], in particular nickel, cobalt, tungsten, molybdenum, titanium, aluminium, copper and iron and their alloys, as well as oxide and nitride ceramics, glass, intermetallic phases and composite materials. Process conditions depend on the type of sintered materials. This process is carried out by filling, closing and venting the tank, heating and increasing the gas pressure in the tank, heating under constant pressure and rapid cooling at a pressure drop at a controlled speed of up to 100°C/min. Hot isostatic pressing (HIP) is used in the aerospace industry, among others for the production of turbine blades, in the automotive industry, e.g. for turbochargers and valves, in the medical industry, among others in manufacturing processes, prosthetic devices and in the petroleum industry for valve bodies.

Powder forging (PF) technology is based on conventional perform cold pressing with their subsequent heating in a furnace with a controlled inert atmosphere and forging with one blow, ensuring complete compaction of the material [69,70]. The process could be realized alternatively by the hot upsetting of the perform with the lateral flow of material or hot re-pressing with the flow of material towards deformation. That technology was developed almost over 20 years ago for the manufacture of diesel connecting rods. Compared to the traditional forging of solid micro-alloy steel, the cost of connecting rods using the Fe – 1.8 Cu – 0.4 powder mix is reduced by approx. 10% while reducing the weight of the connecting rods. There is also a reduction in the noise level during production, reduced vibration and better surface roughness, but the fatigue strength of the connecting rod thus produced is reduced by about 15% [69,70]. This technology was also used to produce engine pistons made from an Al – 4.5 Cu – 0.5 Mg – 0.7 Si powder mixture.

5. Spark Plasma Sintering

Spark Plasma Sintering (SPS) involves uniaxial deformation of the metal and alloy powder charge in an electrically conductive matrix by direct heating of the charge under atmospheric pressure with high heating and cooling speed through pulsating electric current. The current also passes through the charge for its rapid consolidation [64,65, 71-97]. That technology is characterized by the production of a local electric discharge plasma, a combination of the impact of external force fields and electric field on the compaction of the charge and the formation of phases in the particulate system and surface layers, as well as significant heating and cooling rates of the charge and the associated significant temperature gradient. This technology has received various names in the literature, in particular Electric Current Assisted Sintering (ECAS) [81], Current Activated Pressure Assisted Densification (CAPAD) [82], Pulsed Electric Current Sintering (PECS) [83], Field Assisted Sintering Technique (FAST) [84,85], Direct Hot Pressing (DHP) [86,87], Electro Sinter Forging (ESF) [88], Field-Assisted Hot Pressing (FAHP) [89], Plasma Assisted Sintering (PAS) [90], Electric Pulse Assisted Consolidation (EPAC) [91], basically not differing at all or showing slight design differences in the devices used. The name SPS is most often used in the literature [92]. The name PECS is the most accurate because plasma formation during sintering has not been well demonstrated [75].

The invention of spark plasma sintering technology is credited to Arthur G. Bloxam in 1906 in the UK [93], who developed the first DC sintering system for manufacturing of electric incandescence lamp filaments from tungsten and molybdenum alloys. George Weintraub and Harold Rush from the USA completed their works in this area more as a century ago in 1912. They developed the sintering process for refractory materials by applying pressure and electric current. In 1922 Alexander Duval d'Adrian American French used electric-assisted sintering to manufacture parts from refractory materials. In 1930 George F. Taylor from the USA was developing a device for hot-assisted pressing of the composite of tungsten carbide with cobalt [93]. It was not until the early 1990s that industrial interest in this technology increased. In the last two decades, new advanced technologies were developed using the idea of spark plasma sintering [98]. This technology is gaining more and more importance [78,94,95].

Figure 10 shows the SPS sintering device with a vertical uniaxial pressure boosting device with a built-in water-cooled energizing mechanism, water-cooled vacuum chamber, accessories for controlling the vacuum atmosphere and exhaust system, DC pulse generator and control system.

Powder materials are placed between the punch and the matrix between the electrodes, which ensures a rapid increase in temperature to 1000-2500°C [98]. The heating rate is usually 100-1000°C/min, and the externally applied pressure often reaches 30-200 MPa [94]. Initially introduced devices allowed sintering of electrically conductive powders only in electric density compaction (EDC) or electric density sintering (EDS) processes. Non-conductive materials can be sintered in plasma-activated sintering (PAS) and spark plasma sintering (SPS) processes in addition to conductive powders. The PAS process uses a single pulse cycle process that uses pulsating direct current at room temperature for a short time, followed by direct current in the rest of the process. Pulse heating with electric current is used repeatedly during the entire sintering process in a multiple pulse cycle process corresponding to SPS technology. The temperature gradient inside the manufactured element is reduced to achieve sintering homogeneity, the distribution of which depends on the electrical conductivity of the powder, the thickness of the matrix wall and the presence of graphite insulation, preventing direct contact with the workpiece, as well as ensuring the electrical connection between all elements. As a result of the use of a conductive matrix and the use of conductive metal powders, the workpiece is heated quickly. That becomes particularly important when the forms have a large diameter and relatively small height. Due to the possibility of using high heating rates, the SPS technology is particularly useful for materials that can stay for a short time at a high temperature. Such materials can be sintered to achieve a final density with near-net-shape NNS, and therefore it is not necessary to perform their final machining, especially since due to the high hardness of many of them, such processing causes many difficulties.

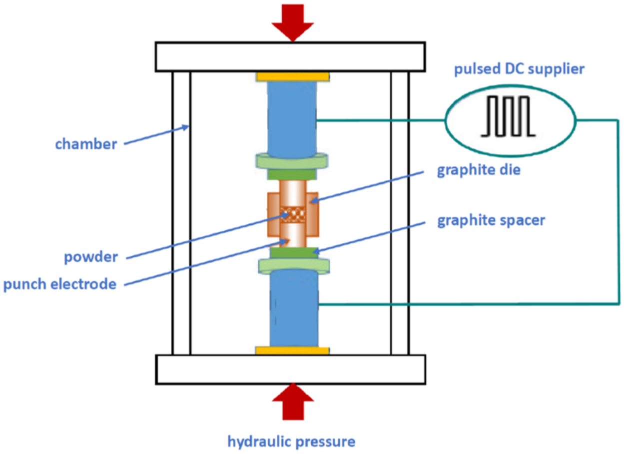

Fig. 10. The principle of spark plasma sintering (SPS)

The SPS technology can be considered as an extension of the hot pressing (HP) method or also electrical resistance sintering (ERS) or can even be treated as a hybrid of many methods [92]. The main difference between SPS and conventional hot pressing methods is that in the SPS method, the current is applied repeatedly in the form of very short pulses, each in less than a millisecond (ms), instead of a single pulse for a longer time, as in the HP method. Therefore, both pressure and temperature are lower than would be required for most other methods in which both factors interact, and the combined heating time is also correspondingly shorter. As a result, less grain growth occurs, and better properties are obtained [92]. SPS technology is considered better than HP or HIP methods [95] due to the improvement of material properties.

SPS technology is considered better than HP or HIP methods [95] due to the improvement of material properties. The obvious benefits include depending on the materials used, improved mechanical properties [99,100], remarkable increase in ceramic's superplasticity [100], improved magnetic [102], electrical [103,104] and thermoelectric [75] properties, higher permeability in ferroelectrics [105], as well as improvement of oxidation and corrosion resistance [100,102] and improvement of optical transmission [106]. Important reasons for such effects include cleaning the grain boundaries in sintered ceramic materials [103,107] and reduced segregation of impurities at the grain boundaries [103] and the associated improvement in binding quality [108]. Clear benefits of using this method over conventional consolidation techniques have been shown, associated with increasing the sintering rate by obtaining full compaction at a low pore volume and at the same time preventing grain growth [75]. In work [109], the characteristics of spark plasma sintering SPS technology were compared with the corresponding properties of HP hot pressing technology (Tab. 2).

Due to the obtained structure and properties of materials, SPS technology can also be an alternative to HIP and MIM technology, although sintering with the participation of the liquid phase is excluded [92]. SPS is a technology considered energy-saving due to the short process time and several processing steps. It is an innovative sintering process and is characterized by low energy consumption from 20 to 35% compared to pressure less sintering (PLS), hot press (HP) sintering, and hot isostatic pressing (HIP) [72,92,109]. In work [75], based on literature data, a wide set of engineering materials is presented, for which spark plasma sintering technology is used on an industrial or laboratory scale (Tab. 3). This demonstrates the very significant development and importance of this technology.

Compared with the methods of hot pressing (HP) and hot isostatic pressing (HIP) or treatment in furnaces with a protective atmosphere, the SPS method provides easy operation and high reliability. SPS technology guarantees accurate control of sintering energy when sintering temperature is reached by heating within a few minutes, depending on the type of material, dimensions and shape of the workpiece. It depends also on the power and type of device. In conventional methods, sintering takes more than ten or even several dozen hours. SPS technology is used in nanotechnology [96], in the processing of ferroelectric ceramics and biomaterials [110] and titanium matrix composites TMC [111]. It was pointed out that the method of SPS can be used to manufacturing titanium sheet by pressing hot-rolled mouldings prepared with this technology [112].

SPS technology has recently been used, among others, for the production of spray discs and high-performance ceramic components such as boron carbide, titanium diboron and sialon. This technology is becoming more and more important in the friction materials sector, for the production of sintered brake shoes used in fast trains and various wagons, as well as in devices using wind energy, and even in quads and mountain bikes. Sintered clutch discs produced in this technology are mainly used for heavy trucks, ships, tractors and other agricultural machinery. The SPS method allows the production of materials with a maximum density and the lowest possible grain size, which is particularly positive in the case of nanostructural processes requiring the preservation of nano functions, despite the need to consolidate powders [72,92].

Table 2.
Comparison of characteristics of technologies SPS and HP

No.	Characteristic		SPS technology	HP technology	No.	Characteristic	SPS technology	HP technology
1	Temperature gradient sintering		○	◕	7	Homogeneous sintering	◔	◔
2	Temperature rise rate		○	◕	8	Expandability	○	◑
3	Sintering time	Temperature rise time	Fast	Slow	9	Productivity	○	◑
4		Holding time	Short	Long	10	Investment in equipment	◔	◑
5	Grain boundary controlled sintering		○	◕	11	Running cost	○	◑
6	Fine crystalline structure controlled sintering		○	◕				

Symbol: ○ very good, ◔ good, ◑ fair, ◕ difficult.

Table 3.
A set of engineering materials for which spark plasma sintering technology is used on an industrial or laboratory scale

No.	Materials groups	Used materials	No.	Materials groups	Used materials
1	Armor materials	Al_2O_3; $(TiB_2)_{0.95}Ti_{0.05}$	17	Laminates	SiC/TiC; Si_3N_4-SiC/BN-Al_2O_3
2	Biomaterials	Hydroxyapatite	18	Machinable ceramics	Si_3N_4/BN; Al_2O_3/Ti_3SiC_2
3	Catalysts	Ni-Mo	19	Magnetic	Nd-Fe-B; Nd-Fe-Co-V-B
4	Cellular structures	Ni-P alloy	20	Nanostructures	ZrO_2; Ti-Al-B
5	Cemented carbides	WC-Co	21	Nanotubes	Carbon
6	Composites	TiB_2-TiC; AlN-TiB_2	22	Phosphors	ZnS family
7	Dielectrics	$Ba_{0.5}Sr_{0.5}TiO_3$; $Ba_{6-3x}\,Sm_{8+2x}\,Ti_{18}O_{54}$	23	Piezoelectrics	K-Na niobate; Sr-Ca-Na niobate
8	Electroceramics	ZrO_2	24	Porous materials	Al; $FeSi_2$
9	Electrode materials	Ni-Mo	25	Pyroelectrics	Pb zirconate-Pb titanate-Pb
10	Ferrites	Mn-Zn, Ni-Zn	26	Shape memory materials	Cu-Zn-Al; NiTi
11	Ferroelectrics	$BaTiO_3$; $NaNbO_3$	27	Solid solutions	TiB_2-WB_2-CrB_2; TiB_2-WB_2
12	Funnctional Gradient Materials FGMs	Nb_5Si_3/Nb; Ti-Si-C	28	Sputter targets	In-Sn oxide; Pb titanate
13	Glassy materials	Fe-Co-Nd-Dy-B; Fe-Co-Ga-P-C-B	29	Superconductors	MgB_2; Nb_3Al
14	Hard materials	WC, SiC, B_4C	30	Synroc	Titanate ceramics
15	Intermetallics	$NbAl_3$; FeAl; NiTi	31	Thermoelectric	$(Bi,Sb)_2Te_3$; Mg_2Si
16	Joining of materials	Mo/$CoSb_3$			

As the cited data and information on high application possibilities indicate, the SPS technology is increasingly used [32,64,65,78,94,95,113] with the proviso that only sintering with the solid phase is possible. The report [93] states that the total market for devices enabling the use of current, vacuum and electromagnetic radiation-assisted sintering technologies covered by the Spark Plasma Sintering (SPS) technology. It is estimated to increase from USD 915 million in 2017 to about 1.3 billion USD in 2022 with a compound annual growth rate (CAGR) of 6.6% in 2017-2022. Electromagnetic radiation-assisted sintering technologies will increase from USD 647 million in 2017 to approx. USD 850 million in 2022 with a CAGR of 5.6% in 2017-2022, while the vacuum-assisted sintering market will increase from approx. 184 million USD in 2017 to around 260 million USD in 2022 with a CAGR of 7.2% in 2017-2022.

The projected progress faces specific development barriers resulting from the increase of the scale of technology from the laboratory to industrial, especially in relation to the production, supply and processing of advanced and technical ceramics [114]. The market continually requires more abundant and larger ceramic blanks, including electrically conductive composites based on ZrO_2 and Al_2O_3, with particular emphasis on ZrO_2-WC/TiN and Al_2O_3-Ni-TiC-SiC with a diameter greater than 200 mm. Such products show great potential in engineering applications, including as components of industrial machines, cutting tools, wearing parts, custom accessories. For their production, it is necessary to use new innovative technology together with appropriate production equipment, including the use of innovative pressure-assisted hybrid heating sintering using a large-scale spark plasma hybridized with an induction sintering device. In May 2015, it was reported that the first binderless element with a diameter of 400 mm was manufactured with the mentioned hybrid plasma sintering technology using the most massive hybrid SPS device available in the world [114]. This device implements hybrid SPS technology in which Joule heating of ceramic powder is combined with induction heating of a graphite matrix as an additional way to provide the necessary heat. It gives the possibility to obtain a homogeneous temperature distribution in a mechanically loaded punch/powder on a large scale/die set-up [114].

Microwave radiation can also be used to sinter preformed parts. Microwave sintering uses microwaves, developing both electric and magnetic fields. The heating is fast and short, which ensures a higher density than conventional sintering. The desired properties are achieved due to the shorter sintering time and the lower sintering temperature required, which, however, may even reach 2000°C due to the design reasons of the devices [92]. Microwave sintering was first developed for ceramics [115], including cemented carbides, and then they were also used for metals [116].

6. Powder Injection Moulding

Powder injection moulding (PIM), often in a variety of metal injection moulding (MIM) is a technology used for mass-produced products with a highly developed surface and relatively small dimensions. It has caused a very dynamic increase in interest in this technology in recent years. The difference between the two methods is that the PIM method uses different powders, and the MIM version uses only metal powders and their alloys. It was assumed that the beginnings of powder injection moulding technology date back to 1849 when pressure casting of non-ferrous metal alloys was introduced [117]. Further development was related to mastering the technology of processing polymer materials and implementation of the first piston injection moulding machines in the USA and Germany. One of its first PIM applications over 80 years ago, back in 1937, was the manufacture of spark plug insulators [118]. The PIM method was used in the 1970s to produce metals and ceramics in electronics, while in Germany in the 1980s, orthodontic components were manufactured by this method [119]. Both the PIM and MIM methods enable the production of products with complex geometric features in the near net shape standard, and due to the high investment costs of the devices used, mainly for high-volume and mass production [120-122]. For this reason, the PIM and MIM processes are not suitable for the production of large parts [123,124]. It is most often used for the production of minimal components, including miniaturized devices [117,125,126], usually weighing no more than 0.5 g and difficult to manufacturing with other technologies, e.g. orthodontic components [127].

Despite the observed development continuously [16,46, 117,119,123,124, 127-147], especially in North America, Germany, Japan and China, none of the PIM and MIM technologies varieties is as developed as classical pressing and sintering. It is evidenced by the relatively small proportion of powders used for this purpose. For example, in 2013, about 365 thousand tons of iron and steel powders were produced in the United States. 90% of them are powders used for powder metallurgy. The example of powders used in powder metallurgy in Europe indicates that only 1% of total powders production is used in the MIM process. Nevertheless, sales of components manufactured by this method increased sharply, e.g. in Europe in 2003-2013 from less than EUR 150 million to over EUR 250 million, and continues to grow in subsequent years. Metallic or ceramic powders, or even mixtures thereof, are used as a powder injection moulding raw material.

In work [148] selected advantages of PIM powder injection moulding technology are presented in comparison with the general disadvantages and advantages of other near net shape materials processing technologies commonly used in the industry for the manufacture of products, including conventional powder metallurgy (Tab. 4). Table 5, on the other hand, compares various features specific to different technological processes of powder metallurgy [149].

Almost from the beginning of the widespread use of MIM technology, the removal of the binder created application restrictions, because it is technologically challenging and time-consuming and is the cause of distortions, gas bubbles and cracks as indelible defects in any subsequent technological operations. Controlled pyrolysis process, based on thermal degradation of the binder, was often used in the absence of oxygen for debinding. The development of this technology has recently been decided by solutions in this area that are alternative, fast and safe for both the environment and the manufactured products. Methods of solvent degradation in a vacuum, under elevated or supercritical pressure, were successively introduced, but the breakthrough was the method of catalytic degradation, and granulates and debinding devices developed on its basis [144,147,150-153].

Table 4.
Selected features of elements manufactured with different technologies

Selected features	Materials processing technologies			
	Powder Injection Moulding	Conventional powder metallurgy	Casting	Machining
Density, %	98	90	95–99	100
Wall thickness, mm	0.1-10	2	5	2
Mass of manufactured elements, g-kg	0.003-17 k	0.1-10 k	> 1	> 0.1
The complexity of technology	◔	◕	◑	◔
Production scale	○	○	◕	◕
Strength compared to solid material, %	100	70	98	100
Surface roughness, μm	0.4-0.8	2	2	0.4-2

Symbols: ○ very high, ◔ high, ◑ medium, ◕ low, ● very low

Table 5.
Comparison of powder metallurgy processes

Characteristic / PM process	Shape complexity	Dimension	Dimensional tolerances	Density	Production yield	Cost
Powder Injection Moulding	○	◕	◑	◔	◑	◑
Hot Isostatic Pressing	◔	○	●	○	●	●
Powder Forging	◑	◑	◔	○	○	◔
Conventional powder metallurgy	◑	◑	○	◕	○	○

Symbols: ○ very high, ◔ high, ◑ medium, ◕ small, ● very small

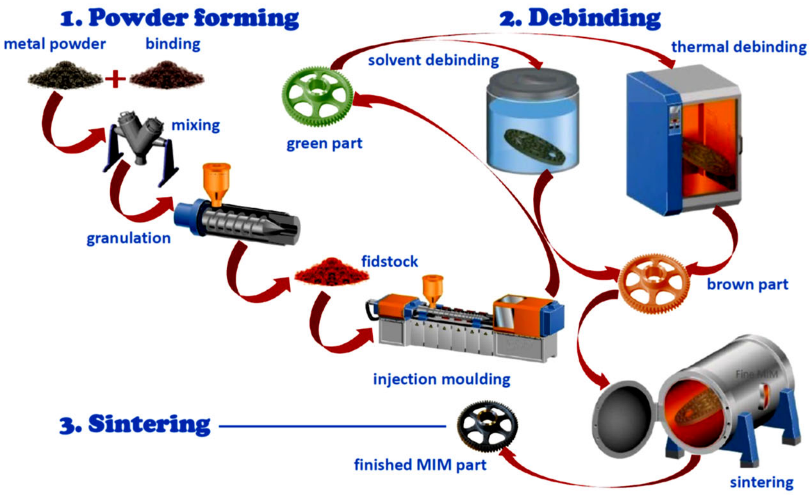

Fig. 11. Diagram of the sequence of technological operations of the metal injection moulding MIM process

Binders are usually mixtures of organic compounds, mainly natural waxes or synthetic polymers, including thermoplastic binders (containing, among others, paraffin, beeswax, vegetable oil, peanut oil, antipyrine acetanilide, naphthalene, PEG), polyacetal (containing polyoxymethylene), gelatin (containing water). The most common are two types of binders based on paraffin and polymer materials, respectively, and a water solution of methylcellulose (Wiech's or Rivers's solution) [117]. The use of thermoplastic polymers as a binder binding metallic or ceramic powder enables its transport and forming in the injector seat. The ratio of powder to binder is closely related to the shape, size of the powder particles, type of material, wettability of the powders through the binder and the properties of the binder itself. It is also related to the conditions for the preparation of the mixture [140,142]. As a rule, it is more challenging to mix ceramic powders with a binder and inject them than metallic powders. To do this is necessary to use a binder's larger fraction. The selection of the binder affects the strength of the green parts and the fluidity of the powder mixture. Mixing is often carried out at elevated temperatures. The entire surface of all powder particles should be evenly coated with binder without particle agglomeration effects. Wetting can be improved by adding surfactants. The binder and other additives must not react with the powder material, nor be damaged during the process. The possibility of recycling the unused portion of the binder and the possibility of reusing equipment and instrumentation should be provided. Removing the binder from the compact must be relatively easy since this operation is highly cost-effective.

The MIM process consists of a sequence of technological operations, including powder forming, debinding and sintering (Fig. 11). Subsequent operations may include machining, hardening, deburring or surface treatment. Injection moulding for MIM is similar to injection moulding of polymeric materials. Despite the similarity of technology and technological devices, considerably higher strength requirements and resistance to abrasion should be taken into account, due to the use of metal powders of higher hardness and usually higher injection pressure. The forming process is only for shaping, while the final properties are shaped only in subsequent operations.

Moulding conditions strongly depend on the type of mould and the geometrical characteristics of the manufactured product, which determine the distortion, flow lines, connection lines, powder and binder separation and final product dimensions. Then the binder is removed by debinding, which is associated with the occurrence of porosity in places previously occupied by the binder, and the preform becomes very fragile. Reducing atmospheres composed of hydrogen with a hydrogen concentration of approx. 85% allows for the highest strength. The preform density after complete binder removal is about 60% of the theoretical density. This density depends significantly on the binder fraction, which can be reduced by up to 30%, and in the case of ceramic powders up to 55%, due to the appropriately selected spherical shape of metal powders and their good wettability. The debinding operation is used to prepare the sintering element. The use of a solvent allows the removal of soluble components of the binder and takes place at room temperature or slightly higher, which facilitates the use of this method. During thermal evaporation, the workpieces are subjected to oven temperature, and the polymer binder is evaporated and extracted as a result of an inert or reducing gas flow through the oven or the application of a vacuum. Catalytic decomposition of the binder requires the use of nitric acid or oxalic acid gas. The mixed-method is associated with combined degradation techniques, e.g. solvent and thermal techniques. For extremely small preforms, for example in the µPIM process, debinding can be combined with heating to sintering temperature [117,126].

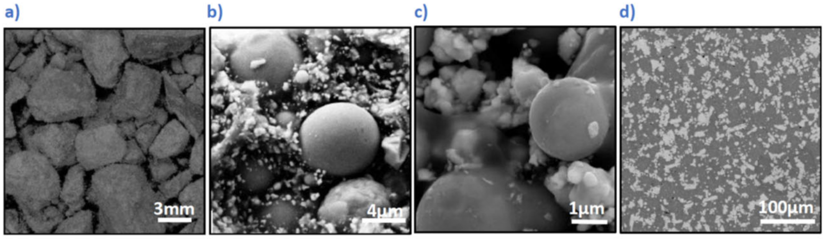

Fig. 12. Structure of carbide-steel HS6-5-2 + carbides a) mixtures of steel powder, carbides, paraffin and polypropylene in the form of granules for injection moulding; b) after dissolving the binder in heptane at 25°C for 1 h; c) after thermal removal of the binder at 400°C for 1 h; d) final product (SEM)

The debinding technological errors include warping, gas bubbles and cracks. Sintering is another technological operation, accompanied by shrinkage of elements from 12 to 20% (usually 15-18%). The shrinkage is strong, which depends on the porosity and can lead to an uncontrolled change in the shape of the sintered parts. Since the change in volume of the sintered material is inversely proportional to its density after forming, an increase in the density of the preform reduces the risk of defects in the shape of the formed preform. Injection pressure, powder size and binder fraction increase the preform density. Sintering is usually the last operation of the technological process that determines the density and properties of the finished product [124]. During sintering, thermal diffusion occurs and, as a result, the workpiece solidifies, which ensures the expected high functional properties [124,127, 154-156]. Because very fine powders are used in the MIM process, the resulting sintered material has a high density. The density of components manufactured by the MIM method is usually greater than 96% of the theoretical density of the material used.

Sintering can take place in high-temperature furnaces using process gas or in a vacuum. Usually, the sintering temperature is from 1200 to 1400°C depending on the chemical composition of the sintered alloy. The properties of materials produced by the MIM method, especially tools, are improved by the release of carbonitrides as a result of the nitrogen-enriched atmosphere during sintering [157]. Sintering, similarly to debinding, can be carried out discontinuously or continuously. Since no additional machining is required after sintering, which is the main advantage of this technology, it is particularly suitable for the production of hard materials, including tools [120,123,158,159], metal tools with relatively high ductility, ceramic tools with high hardness or metal matrix composites (MMC) and ceramic matrix composites (CMC) [136,160-162], as well as high-speed steels, carbide-steels (Fig. 12) and cemented carbides [154,163,164].

It should be noted that the PIM and MIM methods allow the manufacturing of Functionally Gradient Materials (FGM), especially Tool Gradient Materials (TGM) [161,165, 166], which is only possible to obtain by sintering [167] or sequential injection and thermal spraying of coatings [168,169] as well as PVD and CVD methods of coatings deposition on the substrate manufactured by other methods [170]. The authors' own research experience concerns the manufacturing of high-speed steels by the MIM method, as well as tool gradient materials. If the final element should have high mechanical properties, final heat treatment is applied, as in the case of high-speed steels produced by the PIM method [170-174]. Sales of products manufactured by PIM and MIM have been increasing over the past three decades and shows great interest in this technology [18].

Another technology is Mechanical Powder Press (MPP), a process more often called Mechanical Alloying (MA) [19,64, 175-178], which consists in the production of alloys from pristine metal powders by grinding in high power mills. High-energy mills of various types are used, including vibrational, planetary and attritor [32,179]. A mixture of metal powders in appropriate proportions and particles of abrasion-resistant materials, e.g. ceramic materials, cemented carbides or hardened steel, used to crush metal powders, is placed in a sealed mill container. With the use of intense vibrations and centrifugal forces, the charge mixture is ground thanks to the high collision energy, causing the temperature to rise to approx. 200°C (Fig. 13).

The purpose of this action is to obtain the required chemical composition of individual powder particles, and this determines the choice of grinding time. This process was developed in 1966 by the team of John Benjamin [19] as part of research into the production of a nickel-based superalloy for use in gas turbines, summarized in works [180-185], which led to the development of the current process [180].

The grinding mechanism in these conditions consists of multiple and subsequent dynamic welding of powder particles and subsequent crushing. High compressive stresses affect the intense plastic deformation inside the powder particles and generate a metastable structure of mostly nanocrystalline solid solutions, intermetallic phases or mixtures of components. However, it is also possible to produce an amorphous structure. The density of crystal structure defects, mainly dislocations and grain boundaries or low-angle boundaries is constantly increasing, generating repetitive healing and polygonization processes, the annihilation of low-angle boundaries and static recrystallization. Recovery in metal particles with a low melting point is easier, but there is no significant fragmentation of the grains inside the powder particles. Strength and physicochemical properties of the products of this process depend on the type of mill, temperature and milling atmosphere. Figure 14 shows the structure of ground powders or powder mixtures.

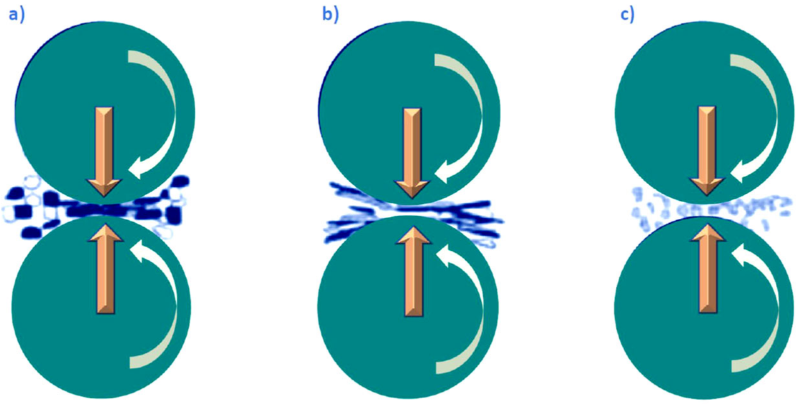

Fig. 13. The principle of mechanical alloying (MA); a) a mixture of input powders: b) elongated composite particles; c) homogeneous equiaxed alloying powder particles

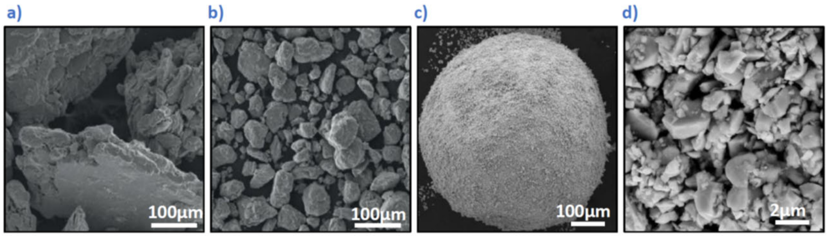

Fig. 14. Structure of ground powders or powder mixtures a) AlMg9Si alloy for 5 h; b) mixtures of AlMg9Si powders and 5 wt.% of polyhedral carbon nanotubes for 20 h; c) granules of a mixture of agglomerated WC carbide and cobalt powders; d) fragment of the granulated surface of agglomerated powders from fig. d (SEM)

The essence of the approach associated with this technology relates to obtaining powders of alloy materials from pure metals, without having to melt them. Mechanical alloying is, therefore, a dry, high-energy ball milling technique that has been used to manufacture a variety of materials. The remaining components of the technological process do not differ significantly from other technological processes of powder metallurgy. Crushed powder in the described process is then subjected to the next consolidation by various methods, ensuring the required shape and form of manufactured products or elements. It can be conventional pressing and sintering, cold or hot pressing of powders, and sometimes hot isostatic pressing (HIP), ensuring even powder compacting and sintering [20]. The manufactured products can be subjected to finishing treatment, and if necessary heat treatment is required to apply, to obtain the desired structure and properties. The use of inert additives to improve mechanical properties at elevated temperatures in metals was first used in 1910 by W.D. Coolidge in tungsten with thorium [180]. It was soon discovered that other micro-additions could also increase creep resistance. In the source work [180], it was written that many useful combinations of metals could not be achieved by melting or by conventional powder metallurgy. Cold-welding metal powders can make such materials in a special high-energy ball mill. With the use of this technology, it is possible manufacture materials outside the range of solubility in the solid-state, as well as those that usually arise at low temperature, new crystalline and quasi-crystalline phases, amorphous phases and ordered and disordered intermetallic phases, as well as among them high-melting nitrides, carbides, borides and oxides [20,177].

The mechanical alloying process for the manufacturing of homogeneous alloy powders (MA), by milling powders of the components of these alloys, is sometimes called mechanical milling (MM) [186]. Mechanical disordering (MD) is sometimes written [187] because as a result of prolonged milling of the intermetallic phase powder, it can pass to disordered state or amorphous phase will occur. Besides, the term of this technology can be found as mechanical attrition [188] or mechanical grinding (MG) [175,176,180], although no grinding shavings are produced as in the machining technology.

A special application of this technology is Mechanical Alloying & Oxides Dispersion-Strengthened (MA/ODS), enabling the dispersion of second phase particles (usually oxides) in the matrix of the produced material using the mechanical alloying approach. For the first time in work [189] concerning sintered aluminium powder (SAP), i.e. sintered aluminium with approx. 20% Al_2O_3, it was shown that it is possible the manufacturing dispersion strengthened materials using powder metallurgy. Approximately 13%, 10-11% and approx. 7% oxide were used in various grades SAP 865, 895 and 930, respectively [190,191]. The strength properties of these materials increase with an increase in the proportion of oxides. Over time, similar technological solutions have become widespread in relation to other alloys, especially high melting alloys, where it is impossible to use matrix oxides of metal, and therefore sufficiently refractory oxides in the matrix of high melting metals, e.g. ThO_2, were used to nickel strengthening by dispersed phases [182]. In work [182], the development of a new dry, high-energy mechanical alloying technological process was announced for the first time, which enables effective dispersion hardening of nickel superalloys by supporting precipitation hardening of the matrix with γ 'particles. The essence of this process is high-energy milling to avoid any surfactant other than air in the high-energy mill container also containing the feed of processed raw materials. Currently, the MA/ODS method can be used to produce materials with different compositions. In addition to aluminium + Al_2O_3, the MA/ODS method produces sinters U + UO_2, Fe + Al_2O_3, Fe-Cr + Al_2O_3, Fe + Fe_2O_3 and Ni + Al_2O_3. The method is used for sinters, e.g. with Ni, Ti and Al matrix, in particular, superalloys MA 754, MA 758, MA 760, MA 6000, MA 956 according to ASTM, containing nickel, and in the case of MA 956 – iron and 15-30% Cr and other additions and micro-additions, in the form of rods, sheets, panels, pipes, wires, sections and forgings [192-195]. Materials manufactured using the MA/ODS method have been used for various elements in various industries, including end caps for diesel engine heaters, thermoelement covers, pallets and their guides, baskets and furnace covers for heat treatment, elements of combustion chambers of coal and oil furnaces, elements of technological equipment for processing liquid glass. In many cases, this technology has become unrivalled.

7. Infiltration

Another hybrid technology combines powder metallurgy, as a result of which a porous structure is intentionally created, enabling the infiltration of a sintered metal intermediate or sintered porous skeleton with metals at a melting point lower than the temperature of the finished sinter [21,196-200]. Various methods may be used, including immersion in molten metal, also at atmospheric pressure and after flushing the chamber with inert gas, or by heating in a sintered atmosphere furnace or a porous skeleton coated with a metal impregnating powder, provided that the temperature is higher than the melting point of this powder. Infiltration methods can be vacuum or at low and high pressure.

Infiltration is a hybrid combination of powder metallurgy methods with casting technology. Certainly, infiltration technology can be considered a particular variant of broadly defined powder metallurgy although its description can also be found in the works on casting metals and their alloys. The pores can also be filled with polymer material, but the analysis of this issue is not within the scope of this paper.

An exemplary structure of composite materials manufactured by the infiltration of sintered porous structures is shown in Figure 15.

The basis of infiltration technology is the production by powders metallurgy methods a porous skeleton with open pores, from metals or metal alloys, as well as often the ceramic skeleton. Various techniques and methods for the preparation of porous ceramics are known, including extrusion [201], reactive bonding [202,203], the addition of pore-forming agents [204,205], polymeric sponge replication [206,207], freeze-drying [208] and foam-gel casting [209-216]. An example of using the infiltration for many years is the production of electrical contacts [217]. Pre-pressed copper powder or forged copper pieces in contact with the porous tungsten preform is heated in a hydrogen atmosphere above the melting point of copper [218-221] and infiltrates the tungsten preform. This preform is made by pressing powder, and the whole process is possible due to the low complexity of the shape, even though it requires high pressure for compaction. As a result, there is very little dimensional change during infiltration, and the volume fraction of copper in the finished product should be initially determined. The input powder should be properly ground, and high sintering temperature is required. Cobalt, nickel or iron are used as sintering activators [222]. Infiltration can be combined with pre-sintering in one thermal cycle [223]. The tungsten content is usually not less than 80% by mass, the preform is compacted at a temperature lower than 1500°C, and in the case of infiltration sintering under these conditions, the preform shows shrinkage of 12-14% and a density above 99% of the theoretical value.

Modern methods for producing porous sintered skeletons useful for infiltration can be classified as follows [138,157,161, 224-228]:

- sintering a mixture of uncompressed or pre-compressed at low pressure or obtaining a spongy structure of a mix of metallic or ceramic powders with the addition or less often without a binder, as the simplest and most common

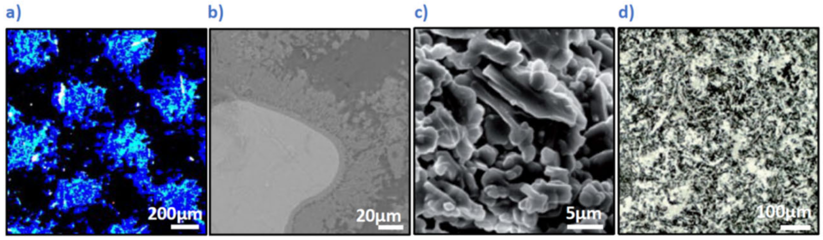

Fig. 15. The structure of composite materials manufactured by infiltration of sintered porous structures; a) porous skeleton manufactured by the selective laser sintering SLS method from pure titanium powder infiltrated with AlSi12 alloy; titanium mapping using EDS (SEM); b) structure as in fig. a light – titanium, grey – transition zone, dark – aluminium alloy matrix (SEM); c) sintered porous Al_2O_3 preform (SEM); d) structure of the preform from fig. c infiltrated with AlSi12 alloy (LM)

sintering method, enabling the production of skeletons with porosity up to 50%;
- sintering or reactive bonding of ceramic fibres or sintering of ceramic and metallic powders with the addition of a pore-forming agent (PFA), as the most allowing the achievement of infiltrated materials with a diverse structure and material fraction forming a porous skeleton according to the principles of percolation; it is used various pore-forming agents (PFA), e.g. polyethene, wax, starch, cellulose, carbon fibres, sawdust [226,228, 229], as well as mineral nanotubes extracted from halloysite and carbon fibres developed based on own work [62, 230-235]; the pore-forming agents are decomposing in high temperature; in a place where voids were formed is introduced the liquid infiltrating metal but only after thermal degradation of pore-forming agents [20,117,236]; as it was demonstrated in the authors' own works [237-240], skeletons can also be made by AM additive manufacturing methods;
- freezing and sublimating the solvent (usually distilled water) from metal or ceramic powder suspensions with a porous structure formed by the volume of the solvent fraction in the suspension [241];
- manufacturing of metallic or ceramic foam from a properly selected mix, through the use of released gases as a result of a chemical reaction or a decomposition reaction occurring at high temperature; it enables the manufacturing of skeletons with porosity up to 90%, but largely with closed porosity and significantly different pore sizes; for this reason, this method is less important in this application.

Then the pores are filled with liquid metal or alloy with a melting point respectively lower than the sintered metal or alloy. The task of the sintered porous skeleton is to strengthen the composite material that will be produced in this way, whose matrix is the metal that infiltrates the pores of the skeleton. The intrinsic property of this porous skeleton is the structure of open, connected pores forming channels that allow the free flow of molten metal.

Infiltration methods fall into three main categories depending on the required pressure for infiltrating liquid metal [242,243]:
- sub-pressure-assisted – vacuum infiltration in which the factor causing infiltration is the excess pressure of molten metal; the sintered skeleton is affected by reduced gas pressure to atmospheric pressure [244];
- pressure less infiltration – infiltration assisted by capillary pressure or pressure less [245] under the action of hydrostatic pressure, including direct infiltration [246];
- assisted with external pressure:
  - low-pressure infiltration, pressure from a few to several hundred Pa to which the gas under the liquid metal is subjected, with the variant of non-pressure infiltration, in which the molten metal penetrates the porous skeleton under the influence of hydrostatic pressure [245, 247-249];
  - pressure/squeeze infiltration, at a pressure of several dozen or several hundred MPa, when in the skeleton pores liquid solidification is progressing after liquid penetrating under the pressure of gas above the metal level [243, 249-251] or done through the piston placed in the press [224-226, 249,252], and alternatively also by centrifugal forces or by Lorentz forces induced by the electromagnetic field.

The porous preform can be saturated with liquid metal throughout the entire pore volume. The structure and properties of composite materials produced by the infiltration of porous sintered skeletons depend on many factors, including from the surface tension and viscosity of the molten metal and the conditions of liquid penetration into the porous structure, including pressure, insertion speed, sintered skeleton and liquid metal temperature, and the possibility of forming composite material after infiltration [253]. It is crucial to ensure the required wetting of the pore surface by the infiltrating liquid metal, which can be improved very often before infiltration [249-251, 254], using one of the possible methods, by [248,253,255]:
- increasing the temperature of the liquid metal or infiltrating alloy;
- the use of an atmosphere to prevent oxidation of the molten metal or infiltrating alloy;
- appropriate selection of the chemical composition of the metal or infiltrating alloy by introducing Si, Ti, Mg, Ni, Cr, Ca, Li alloying additives that affect surface tension changes, adhesion work and contact angle, as well as the type of chemical reactions mainly in the zone of connection of liquid metal with the surface of the porous skeleton and the formation of a new phase in this zone [237]; the strength of the connection is significantly improving when the silicon addition is in an aluminium alloy matrix, as in the case of the combination of $AlSi11/AI_2O_3$ compared to Al/AI_2O_3.

The wettability of the sintered scaffold by liquid metal is essential in infiltration processes with molten metals and alloys [256,257]. It is also strictly dependent on the type and analogous listed above properties of the sintered skeleton. Essential factors affecting wetting include [258] the work of adhesion for separating the unit surface of bodies in contact and creating two new surfaces, the work of cohesion to separate the unit surface of the same body and create two new surfaces of the same phase. The wettability of the liquid infiltrating metal takes into account the contact time and the presence of an oxide coating on the metal drop, which most importantly determines, for example, the wettability in the case of aluminium alloys used as infiltrating materials [257, 259-262].

Wettability in infiltration also depends on the properties of the sintered reinforcing skeleton. It depends on the chemical composition and surface roughness of the skeleton material. However, it can be assumed that if R <10 nm it is an insignificant factor, and porosity above 5-8% by volume reduces the contact angle and makes infiltration difficult. Wettability can only be improved by changing the technological conditions of infiltration and applying thin layers, mainly metallic, on the inner surface of the pores [263,264], including layers of nickel alloy [256, 265-267], Ni-P coating [256,268], as well as copper, iron, platinum. TiO_2 layers can be applied by chemical and physical vapour deposition methods [257,263,268,269].

The properties of the contact zone between the metallic matrix and the reinforcement are essential to transfer loads between them and determine the properties of the composite material produced [251,257,260,262,266]. These connections can be:

- mechanical if the reinforcing elements are mechanically connected to the matrix;
- chemical, related to diffusion processes between the matrix and strengthening;
- adhesive, due to bonds between the adjacent matrix and reinforcement surfaces.

The use of infiltration as a highly cost-effective technology is essential to obtain a wide range of composite materials and brings numerous technological benefits [229, 270-272]. This technology is characterized by high efficiency and can be used in mass production of composite materials with a wide range of reinforcing and matrix chemical compositions. This method can be used to produce elements with a shape similar to a three-dimensional net, alternatively in the entire volume or only locally. These materials attract the special attention of producers, especially in the automotive industry. Technological difficulties with the implementation of this technology are associated with limited wettability inside the pores, depending on the chemical composition of the skeleton, difficulties in degassing the pores and ensuring full dispersion of the matrix reinforcement, due to the complex design features of the sintered porous skeleton, most often in the form of a porous preform [229,265,267,273].

On the other hand, it is relatively easy to recycle composite materials so manufactured, although it is most advantageous to reuse them without separating the components. Separating matrix and porous skeleton materials is more troublesome. It is possible to use mechanical separation methods. For this purpose, it is necessary to heat this material to a temperature that ensures the melting of all matrix material and subsequent squeezing the liquid from the porous skeleton under the influence of pressure. Chemical separation methods consist in using a material stream with higher affinity for the reinforcing phase than for the matrix. As the flux, the material with the lowest solubility in the matrix, e.g. NaCl-KCl-Na_2SiF_6 is used to remove oxide inclusions from liquid aluminium and its alloys [274].

As a special technology should also be mentioned continuous powder processing (CPP). That technology involves rolling powdered material at a speed of 0.5 m/s to reduce the thickness twice and produce a strip, which is then sintered, rolled again and finally sintered ready to get sheet metal. This technology produces sheets and parts in the electrical and electronics industries as well as tapes for making coins [21,65]. Continuous powder sputter on a moving strip cold with subsequent sintering can be used to make tapes, although the problem is to remove produced materials from the movable strip. Continuous powder extrusion (CPE) is made with a binder or plasticizer cold at room temperature with addition, and hot without reinforcement. During the cold extrusion, the metal powder is pressed in the matrix. The extrusion through the hole in the base of the matrix is a result of it (Fig. 9a).

8. Final remarks

In this paper, which is a comprehensive review of the literature the conventional technologies using the powders of metals, their alloys and ceramics are presented. It is a technology that is constantly developing and its importance cannot be overestimated. It was pointed out that among all conventional powder engineering technologies a few are the most promised as follows, mechanical alloying together with Oxides Dispersion-Strengthening, spark plasma sintering, whose range of applications is rapidly expanding and plays the important role, including in nanotechnology, powder injecting moulding, hot isostatic pressing and infiltration, which allows the obtaining of new composites. The ASEA-Sora Process technology enabling the production of large-size blocks, billets and rods is one of the constantly developing technologies, although it is discussed in another paper within this series of papers. Powder engineering is an important technological area that really co-decides about the development of industry and economy at the Industry 4.0 stage. Hence, this scope should be included in the Industry 4.0 development model.

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