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NEW RESEARCH DIRECTIONS IN POWDER METALLURGY

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Abstract. An overview on the present research and development directions in Powder Metallurgy (PM) is realized. There are analysed investigations focused on the mechanical properties of sintered structural parts improving, for overcoming the PM limitations concerning the complex shape and large parts realization as well as for the advanced materials obtaining, like nanocrystalline materials, intermetallics and composites, the most recent methods for this purpose being considered.

Keywords: Advanced Technologies, Powder Metallurgy, Advanced materials, Powders, Sintered structural parts, Powder Injection Molding, Mechanical Alloying, Reactive Processing.

1. INTRODUCTION

Over the last years Powder Metallurgy (PM) has known an impressive development [1]…[3]. This is eloquently highlighted by the upgraded classification of its present applications given in Figure 1. The key factors of this development are its high potential in advanced material producing (some of them impossible to be produced by other technological methods), its known advantages in structural parts production [4], as well as the less influence of the PM companies' activity than of the traditional, especially metallurgical, ones on the ecological system [5]. As PM applications cover very diversified domains of the modern industry, their development continuously stimulate the research efforts for PM progress. The most important directions of these intensive researches will be analysed in this paper.

2. SINTERED PART TECHNOLOGY IMPROVING

Sintered parts have applications both as structural parts and as parts realized from special materials (see Fig.1). For both, improving the processing technologies to overcome the known PM limitations concerning high mechanical properties, complex shape and large parts realization are well-defined research targets.

2.1. Researches for mechanical properties improving

The largest PM application from the tonnage point of view is to the structural parts, especially for the automotive industry, production [1]. They are commonly made of low-alloyed with C, Cu, Ni, Mo and, recently, also with Cr, Mn, Si steels. There are several research directions for their mechanical properties improving.

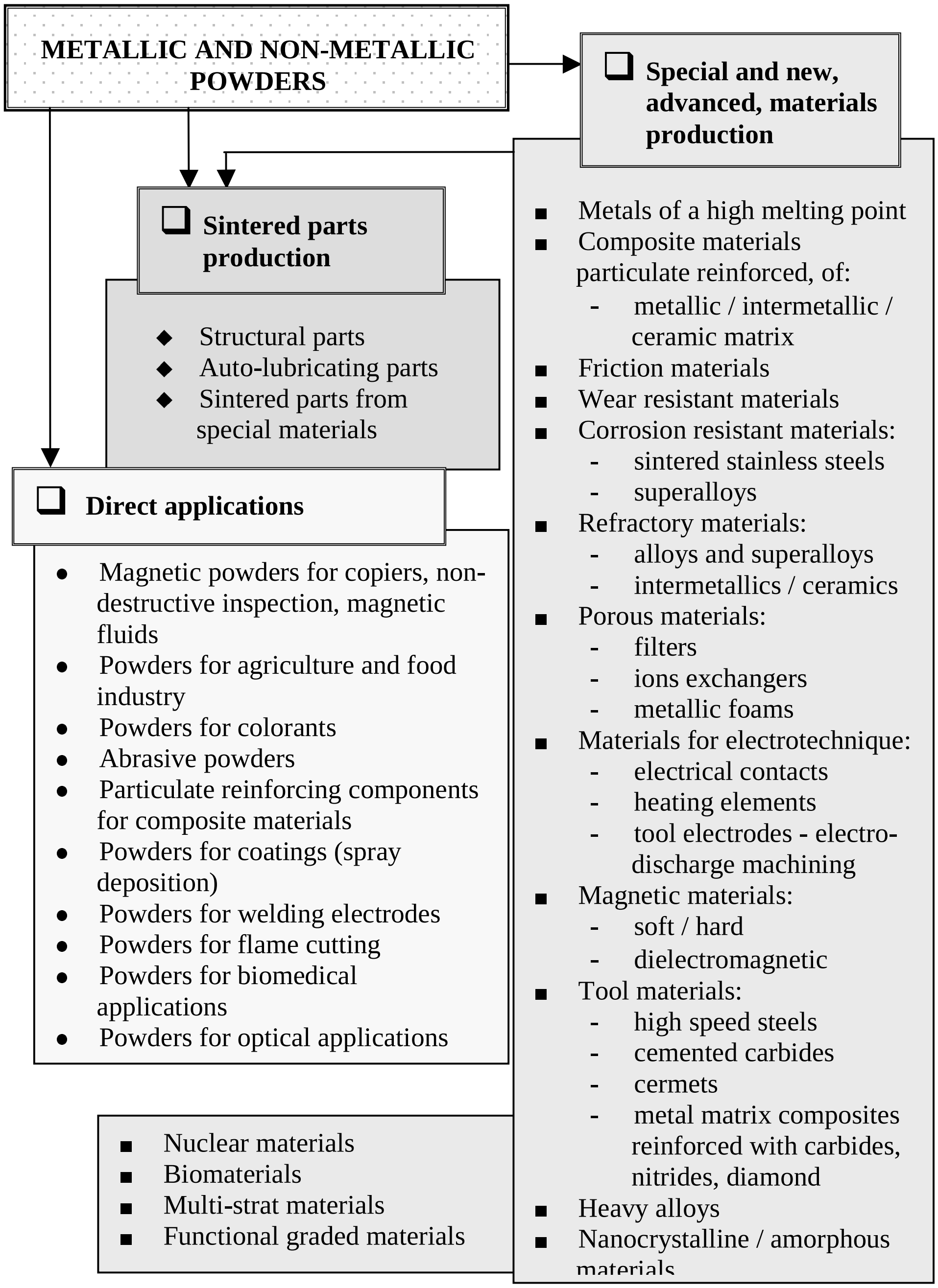
2.1.1. Increasing powder compressibility/homogeneity, dust and hazard reduction

In the sintered structural parts production, water atomized iron powders are going to replace the sponge ones [6]. Therefore, numerous researches are focussed on the atomization technology improvement for a better particle size distribution and a higher cleanliness obtaining. Beside those concerning the atomisation nozzle geometry and processing parameters of standard units optimisation [7], high-pressure water atomization (up to 150 MPa) is investigated to increase fine particle fraction, enhancing particle size distribution [8]. Researches are too performed to introduce vacuum induction melting, degassing and pouring furnaces to replace the common ones. For an even more advanced refining and formation of ceramic inclusions by the melt contact with ceramic liners avoiding, systems with cooper water-cooled walls - applying the slag triple-melting refining process were recently developed [9]. All are expected to improve powder purity and compressibility.

To enhance the sintered steels homogeneity and to reduce dust, the traditional elemental powder mixtures are being replaced, in spite of their lowest cost. As alloyed atomized powders have a low compressibility, diffusion partially alloyed with Cu, Ni and up to 0.5 % Mo powders, with fine particles of alloying elements diffusion bonded to iron, are used in Europe and Japan, e.g. Distaloy [6] and DF-C [10] grades. For higher Mo contents, due to its low diffusivity in Fe, the double alloying method joining atomisation with diffusion has been recently developed [11]. In America, instead, although similar powders are produced (e.g. Ancorsteel FD [12], Atomet DB [13]), they found a limited market due to the high cost. New multi-component powders, with alloying elements bound to iron particles through special binders/chemical treatments, e.g. Ancorloy [12], are replacing them for the lower price. Finally, intensive researches are done for the hazardous and expensive Ni from these powders replacing by more ecological and cheaper Cr, Mn, Si [1].

2.1.2. Improving the die filling and lubrication processes

A high filling density and uniformity over the cross section of the die cavity is essential for good quality parts obtaining. It strongly depends on the powder flowing rate and, for a given powder, on the wall thickness, type of compacting lubricant, filling shoe movement in respect to wall direction (Fig. 2). Numerous studies, some based on the process modelling, are carried out for its enhancing. Improving the filling system design, contour filling, using of agitating feed-shoes or vibratory feeders, powder fluidization/granulation with an organic binder [14].



METALLIC AND NON-METALLIC POWDERS

❑ **Sintered parts production**

- ◆ Structural parts
- ◆ Auto-lubricating parts
- ◆ Sintered parts from special materials

❑ **Direct applications**

- ● Magnetic powders for copiers, non-destructive inspection, magnetic fluids
- ● Powders for agriculture and food industry
- ● Powders for colorants
- ● Abrasive powders
- ● Particulate reinforcing components for composite materials
- ● Powders for coatings (spray deposition)
- ● Powders for welding electrodes
- ● Powders for flame cutting
- ● Powders for biomedical applications
- ● Powders for optical applications

❑ **Special and new, advanced, materials production**

- ■ Metals of a high melting point
- ■ Composite materials particulate reinforced, of:
  - metallic / intermetallic / ceramic matrix
- ■ Friction materials
- ■ Wear resistant materials
- ■ Corrosion resistant materials:
  - sintered stainless steels
  - superalloys
- ■ Refractory materials:
  - alloys and superalloys
  - intermetallics / ceramics
- ■ Porous materials:
  - filters
  - ions exchangers
  - metallic foams
- ■ Materials for electrotechnique:
  - electrical contacts
  - heating elements
  - tool electrodes - electro-discharge machining
- ■ Magnetic materials:
  - soft / hard
  - dielectromagnetic
- ■ Tool materials:
  - high speed steels
  - cemented carbides
  - cermets
  - metal matrix composites reinforced with carbides, nitrides, diamond
- ■ Heavy alloys
- ■ Nanocrystalline / amorphous materials

- ■ Nuclear materials
- ■ Biomaterials
- ■ Multi-strat materials
- ■ Functional graded materials

Fig. 1 - Tentative classification of the present PM applications.

are several proposed solutions. The most innovative seems to be powder fluidization by a current of gas of a low pressure introducing upstream in the filling system. It creates frictionless walls–powder / powder particles interfaces. A uniform powder flow results, eliminating the influences of variations in powder characteristics/ambient conditions.

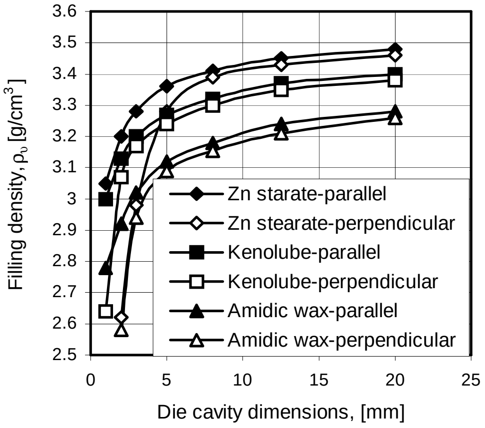


Fig.2 - Filling density distribution as a function on the die cavity dimensions and filling direction [14].

On the other hand, a good lubrication during compaction plays a major role in obtaining a high and uniform density. Proportion and type of compaction lubricant influence too the possibilities of its elimination prior to sintering. For all types of lubricants, a proportion of over ~ 0.8 % leads to the powder compressibility reducing [4]. To enhance lubrication and reduce the lubricant proportion, beside the classic Zn/Li stearate / amidic wax, new multi-component lubricants (Kenolube, Metallub etc.) were created [6], some allowing a content of only 0.3 % [11]. However, maximum compacting density seems to be obtained for die wall lubrication (Fig. 3), higher than for mixed-in-powder lubricants [15]. Even special devices are produced for this purpose.

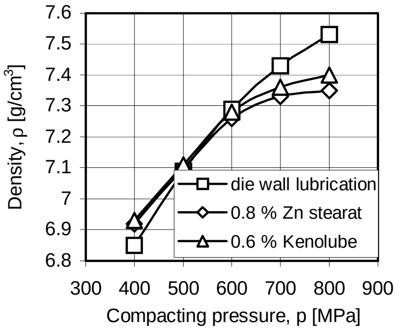


Fig. 3 - Compressibility curves of ABC 100.30 iron powder for different lubrication systems [11].

2.1.3. Advanced compaction methods

To improve densification without productivity reducing, two new compaction methods are being developed: warm compaction and high velocity compaction.

2.1.3.1. Warm compaction

Warm compaction is based on the notable decreasing of yield strength of the ferrite steels at heating (Fig. 4). Consequently, deformability of particles and implicitly, compressibility of such powders notable increases even at a moderate heating (up to 150 ^{0}C), allowing a higher densification at the same applied pressure. A lubricant resistant at these temperatures, able to assure a good lubrication, without to decrease the powder flowing capacity, and also a heating system of both powder and tooling is necessary, instead. Several companies developed such lubricants and heating systems (Fig. 5). Powders for warm compaction are usually delivered previously mixed with lubricant (e.g.

Densmix [11]), assuring, at 150 ^{0}C and 700 MPa a porosity below ~3 [%]. Investigations are performed to decrease the working temperature for the same densities obtaining, in order to improve die filling / increase tool life. Such a lubricant, working at 120 ^{0}C, was recently reported [16].

2.1.3.2. High velocity compaction

Although compaction by explosion (shock compaction) has been used for several years, it was not extended due to its difficulties and low productivity. The dynamic compaction idea was recently reconsidered; new technological variants, less dangerous and of a high productivity, are being developed. So, Combustion Driven Compaction (CDC, Fig. 6) uses the controlled release of energy from combustion of natural gas to compact powder. Another variant, High Velocity Compaction (HVC), realizes densification by intensive repeated shockwaves created by a hydraulically - operated hummer [18]. Both CDC and HVC are able to develop higher loads than the currently used for compaction (Fig. 7), leading to a higher density (at Fe, 7.2÷7.4 [g/cm^3] [17]).

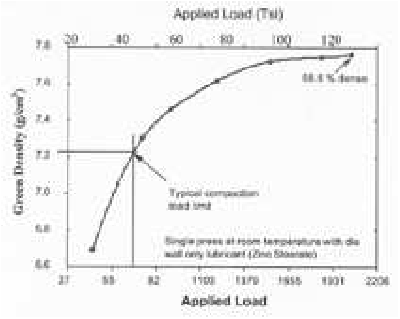


Fig. 7 - Compressibility curve for CDC [17].

Yield strength of ferrite steels variation vs. temperature [16].

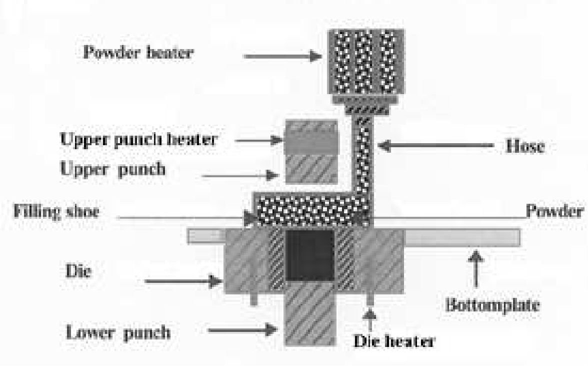


Fig. 5 - Schematisation of a warm compaction system [16].

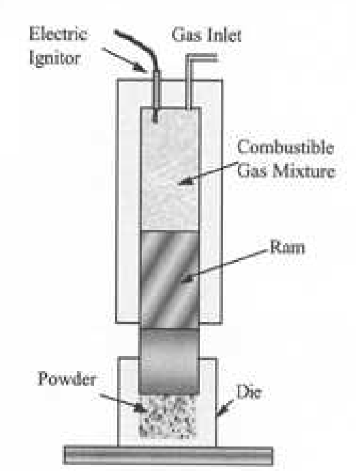


Fig. 6 - Schema of the CDC process [17].

2.1.4. Advanced sintering methods

High temperature sintering and activated sintering are getting an increasing attention.

2.1.4.1. High Temperature Sintering

In High Temperature Sintering (HTS), a temperature with 100 ÷ 160 ^{0}C over the commonly used ones (for ferrous parts 1250 ÷ 1285 ^{0}C) is adopted. On this way, the solid-state sintering mechanisms increase in intensity, resulting in a higher densification and better homogenization, or even in the final sintering stage achieving [4], especially fatigue resistance and toughness being improved (Fig. 8). Although these effects have been known, HTS application, requiring special sintering furnaces equipped with ceramic/carbon-carbon composite muffle and transportation system, was not possible until they could be economically fabricated [19]. Beside its positive effects, HTS leads to the crystal grain growth. To avoid it and for cost reduction, HTS is still investigated.

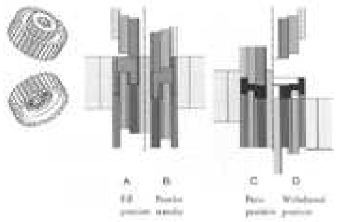


Fig. 9 - Multi-punch die complex part compaction.

2.1.4.2. Activated sintering

Sintering activation, especially by a transient liquid phase creation, is too in attention. It is possible by addition of small amounts of “activators” - forming with the base powder an eutectic of a melting point below the sintering temperature. At ferrous materials, the most used activator is phosphorus (added as Fe_3P)[11]. Some companies are even producing Fe-P powders or premixes [6],[12]. Boron, forming with Fe an eutectic at a lower content than P, caught too recently a high interest [20].

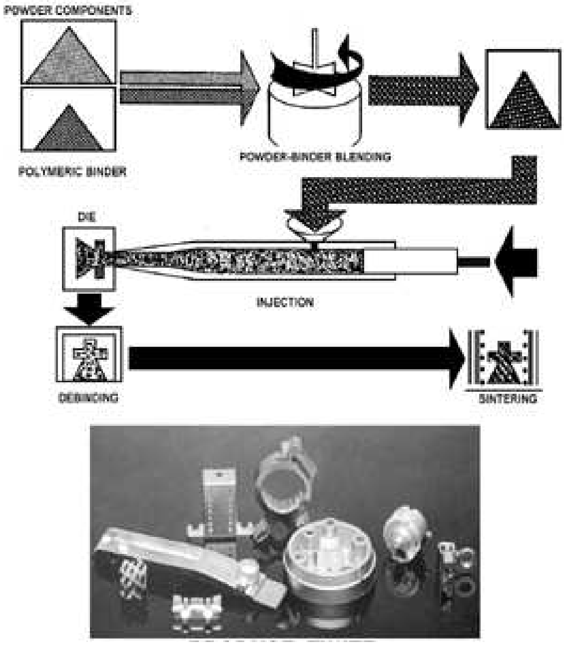


Fig. 10 - PIM process and obtained parts [4].

2.2. Extending PM capability of complex parts processing

Although a notable progress in the complex PM parts production has been achieved by introducing the new computer controlled multi-level presses, realizing the compaction by a multi-punch die with separately operated punches (Fig. 9), as this can compact only linear parts, researches are now focused on the complex 3-D parts realizing, especially by Powder Injection Molding/Selective Laser Sintering.

2.2.1. Powder Injection Molding

Powder Injection Molding (PIM) (Fig. 10) combines the polymer injection technology with powder metallurgy. By injection of a powder-binder mixture (feedstock) in a die cavity, theoretically any shape can be realized. Investigation are presently done to obtain fine spherical powders of an increased flowing capacity, from a higher diversity of materials, to improve binder properties / feedstock rheological behavior for a better die filling, to improve debinding / sintering / mechanical properties, dimensional control [21].

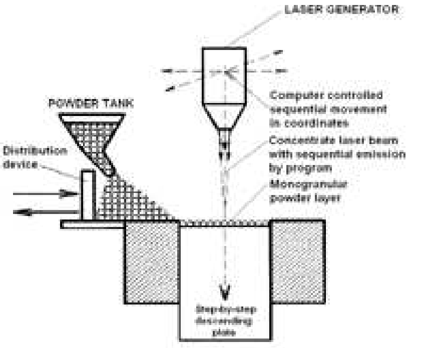


Fig. 11 - Schematic representation of SLS principle.

Fig. 12 - Example of a complex part realized by SLS [22].

2.2.2. Selective Laser Sintering

Selective Laser Sintering (SLS) represents the latest development in the metal powder forming. It consists of monogranular powder layers successively realizing on the machine plate, able to sequentially move down by a step equal to the layer thickness (Fig. 11). In each layer and between two successive layers, a laser beam, by a computer controlled sequential displacement in coordinates, joints particles upon a program to realize the required cross section of the part for that layer. The process is repeated until the whole part is “built”, layer-by-layer. At its end, the un-sintered particles are removed. It is obvious that SLS allows large possibilities concerning the part geometry / dimensions (Fig. 12) and processed materials. However, the obtained mechanical properties are modest

and a supplementary sintering operation is usually necessary. Numerous researches are focused on SLS improving by the laser

beam power/resolution/powder sinteribility increasing (e.g. by coating) [22].

2.3. Large sintered parts realization

Beside the re-consideration of powder forging, determined by technological equipment development through introducing robotized transfer directly from the sintering furnace to the forging press and then to the controlled cooling unit [4], Spray Forming is the most promising method for large PM parts realization.

2.3.1. Spray forming

In Spray Forming (SF, Fig. 13), the stream of droplets formed in a gas atomisation unit is accelerated by the gas jet to the rotating cavity of an open mold (substrate). The adjustable rotation of the mold allows a uniform compaction of the atomized particles. A properly adjusted downward movement of the growing part allows for a permanently constant distance between the atomising nozzle and part, and thus its homogeneous growth. SF has applications to the billets, tubes, rolls and flat parts of high dimensions production [23]. Processing route is much simpler and productive than in common technology, homogeneity is higher. It is still in research, being suitable for alloyed steels/composites processing.

2.4. Improving metal matrix properties

Sinter-hardening and particulate reinforcing are the most known ways of mechanical properties of steel matrix improving. To increase the sinter-hardening efficiency, modular furnaces with a convective cooling zone – by sintering gas re-circulation, were recently realized [24]. Particulate matrix reinforcing was reported e.g. by microalloying technique from classic metallurgy application in PM [25].

3. SPECIAL/ADVANCED MATERIALS PRODUCTION

As results from Figure 1, PM has numerous applications to special/advanced materials production - as sintered parts/coatings. Only the most important, new, of high performance, PM technologies of such materials obtaining will be considered.

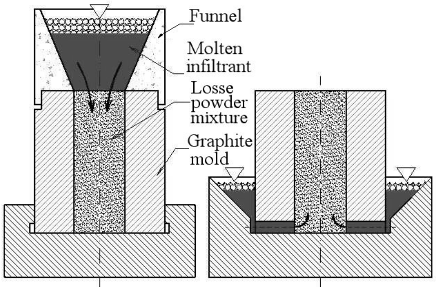


Fig. 14 - SILP principle [26].

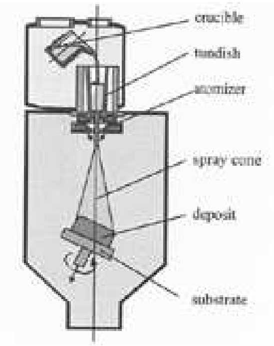


Fig. 13 - Principle of the SF process [23].

3.1. Sintering by infiltration of loose powders

Sintering by Infiltration of Loose Powders (SILP) is a relatively new technology that combines sintering with casting. It consists in a mold cavity filling with a selected mixture of powders, followed by its infiltration with an appropriate molten alloy (Fig. 14) [26]. As a result, a liquid phase sintering process occurs leading to a new alloy formation. The powder mixture may contain some reinforcing components, e.g. carbides, diamond etc., which are diffusion bonded or mechanical embedded by the matrix, resulting a composite of required properties.

SILP allows large and complex parts processing from a broad variety of materials, e.g. diamond drilling tools [26]. Investigation are being performed to enlarge its application, to improve liquid / solid components wetting, to decrease processing temperature for avoiding RCs alteration [27].

4.2. Mechanical Alloying

Mechanical Alloying (MA) is too a relatively new technology that enables a large variety of advanced materials obtaining. It consists of material milling in a high energy mill, process producing repeated powder particle deformation, work hardening, fracturing, clean surfaces forming, local heating, solid state welding,
re-fracturing and so on, under the impact energy transferred to them from collisions of the chaotic moving balls [28]. Pure alloys/solid solutions of an extended solubility limit are obtained if components does not react each other, or if the reached temperature, produced by powder heating as a result of the mechanical energy of collisions partially transformation in heat, is too less for a possible reaction ignition (Fig. 15-c). Intermetallics or other types of compounds are formed when components reacts together or with the milling atmosphere - if the ignition temperature of the reaction, T_{ig}, (a), is reached by the particles (b). Finally, amorphous phases / nanocrystalline materials are obtained by a mechanical disordering at an enough long MA time. In Table 1 are given several examples of such advanced materials obtained by MA.

Table 1
Typical advanced materials produced by MA [28].

Solid solutions				Intermetallic compounds	Amor-phous phases
Solvent	Solute	Equilibrium [% at]	MA [% at.]		
Al	Mg	2.1	23.0	AlCo	Al-Cr
	Mn	0.0	18.5	AlMn	Al-Fe
	Ti	0.0	6.0	AlNi	Al-Ni
Fe	Al	18.5	50.0	$AlNb_2$	Al-Nb
	Si	9.0	27.5	Cr_2Nb	Al-Ti
Ni	Ag	2.0	9.0	Fe_3Si	Ti-Co
	Al	10.0	27.0	FeTi	Ti-Cu
	Nb	6.0	15.0	Mg_2Si	Ti-Ni
Ti	Al	36.0	55.0	NiAl	Zr-Cu
	Mg	2.9	60.0	TiB_2	Zr-Fe
V	Co	7.0	40.0	Al-Ni-Ti	Nb-Cr
Zr	Al	0.5	15.0	Ti-Al-Nb	Nb-Si

Intensive research are being performed for the MA process better understanding, its application to new components / nanocrystalline materials obtaining, for the resulted powder sintering to obtain bulk materials without to alter their microstructure, especially the nanocrystalline one, for the direct bulk MA processing technology and parameters establishement [29].

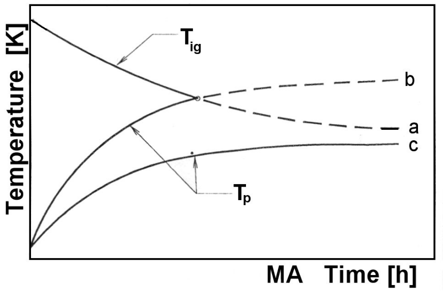


Fig. 15 - Ignition and particle temperature vs. MA time [28].

4.3. Reactive Processing

Reactive processing (RP) is an alternative to conventional methods, suitable to produce advanced materials containing oxides, carbides, nitrides, borides, silicides, intermetallics etc., like bulk materials, metal/intermetallic matrix composites with in-situ synthesised reinforcing components, coating composite layers. It is based on the possibility of some elements to form, by exothermic irreversible reactions, the above mentioned compounds. The thermodynamic condition for this purpose is a negative Standard Gibbs Free Energy of reaction/compound formation, $\Delta G^0_{f/r} < 0$. Such synthesis reactions are highly exothermic, facilitating the bulk materials/coating layers obtaining.

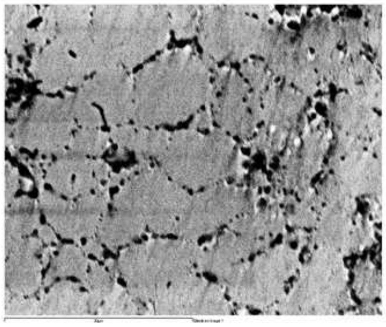


Fig. 16 - SEM image of the in-situ NiAl particulate reinforced with TiB_2 and Al_2O_3 [30].

Although RP can be realised in numerous variants, in the solid (S), solid-liquid (SL) and transient solid-liquid-solid (SLS) states of the reactants, the most important are Self-propagating High-temperature Synthesis - SHS (S), Reactive Infiltration (SL), Reactive Spray Deposition (SLS) [30]. The largest application to the bulk materials obtaining is SHS in the "Pressure assisted simultaneous synthesis" ("thermoexplosion") mode. A typical SEM microstructure of a NiAl matrix composite, realised on this mode is given in Figure 16. Numerous recently published papers on this matter demonstrate the great interest in the RP application for a large variety of special materials, e.g. FGM, obtaining [31].

4. CONCLUSIONS

The above presented developments and future trends in PM demonstrate the large aria of research directions that are still opened for its further development.

REFERENCES

1. D.G., WHITE, State-of-the-North American P/M Industry - 2002. *Advances in Powder Metallurgy and Particulate Materials*, MPIF Ed., USA, 2002, Part 1, p. 1–12.
2. L. ALBANO-MÜLLER, Powder Metallurgy in Europe at the Start of the New Millennium. *Proceedings of 2000 PM World Congress*, Kyoto, Japan, JPMA Ed., 2000, Part 1, p. XVII-XXXI.
3. Y., FUJIWARA, The Present Situation and the Future of PM in Asia and Oceania. *Proceedings of 2000 Powder Metallurgy World Congress*, Kyoto, Japan, JPMA Ed, 2000, Part 1, p. XXXII-XLIV.
4. *** * *** *Materials Handbook*, ASMI, Ohio, vol.7, 1998, 950 p.
5. K. TAKEUCHI, The P/M Materials from the View Point of Environment. *Proceedings of 2000 Powder Metallurgy World Congress*, Kyoto, Japan, JPMA Ed., 2000, Part 1, p. 204-208.
6. * * * *Höganäs Iron and Steel Powders for Sintered Components*. Höganäs AB, 1998, 245 p.
7. J. HAMILL, ET AL., Water Atomized Fine Powder technology. *Proceedings of 2000 Powder Metallurgy World Congress*, Kyoto, Japan, JPMA Ed., 2000, Part 1, p. 367-370.
8. A. LAWLEY, *Atomization*. MPIF, Princeton, New Jersey, USA, 1992, 166 p.
9. S., PLEYER, Innovative Design of Large Scale Metal Powder Production Plants for Advanced Materials. *Proceedings of 2000 PM World Congress*, Japan, JPMA Ed., 2000, Part 1, p. 371-374.
10. * * * *Atomzed Steel Powder*. Kobe Steel Ltd., 2002, 28 p.
11. * * * *Iron and Steel Powders for Sintered Components*. North American Höganäs, 2002, 393 p.
12. * * * *Hoeganaes Iron and Steel Powders*. Riverton, USA, 2002, 8 p.
13. * * * *ATOMET Powder Metallurgy (PM) Products* – Q M P, Canada, 2001, 6 p.
14. M., GUILLOT, Study and Improvement of Die Filling System for Reducing Density Variations. *Advances in PM & Particulate Materials,* MPIF Ed., USA, 2002, Part 4, p. 237-251.
15. M., KONDOH, High Density Powder Compaction Using Die Wall Lubrication. *Advances in Powder Metallurgy and Particulate Materials*, MPIF Ed., USA, 2002, Part 3, p. 47-54.
16. U., ENGSTRÖM, Materials Properties and Processes Robustness Obtained with Warm Compaction Improved DENSMIX Powder. *Advances in Powder Metallurgy and Particulate Materials*, MPIF Ed., USA, 2002, Part 4, p. 4-118-4-129.
17. D.L., KRUCZYNKI ET AL., Driven Combustion Compaction. *Advances in Powder Metallurgy and Particulate Materials*. MPIF Ed., USA, 2002, Part 4, p. 43-52.
18. P., SKOGLUND ET AL., High Density PM Components by High Velocity Compaction. *Advances in Powder Metallurgy and Particulate Materials*. MPIF Ed., USA, 2002, Part 4, p. 85-95.
19. I., CREMER, Complex High-Temperature Sintering Equipment. *Proceedings of 2000 Powder Metallurgy World Congress*, Kyoto, Japan, JPMA Ed., 2000, Part 1, p. 244-247.
20. M., SELECKÁ ET AL., Properties of Liquid Phase Sinterd Fe-Mn-B-C Alloys. *Proceedings of 2000 PM World Congress*, Kyoto, Japan, JPMA ED., 2000, Part.1, p. 16-20.

21. M., MAETZIG, Strategies for Injection Moulding Metals and Ceramics. *Proceedings of the 2002 World Congress on PM & Particulate Materials*, USA, MPIF Ed., 2002, Part 10, p. 10-33.
22. J., KOTILA ET AL., Direct Metal Laser Sintering in 20 µm layer thickness. *Advances in Powder Metallurgy and Particulate Materials.* MPIF Ed., USA, 2002, Part 4, p. 164-170.
23. C., CUI, Production of Homogeneous Bearing Steel Billet by Spray Forming. *Proceedings of the 2002 World Congress on PM & Particulate Materials*, USA, MPIF Ed., 2002, Part 4, p. 201-205.
24. H., WEBER, Sinter-hardening of New PM Steels: Modular Furnace Concept for Stringent Sintering process Conditions. *Advances in Powder Metallurgy and Particulate Materials*. MPIF Ed., USA, 2002, Part 13, p. 96-101.
25. ORBAN, M. and PALFALVI, A. – Some Aspects of the Structural Sintered Steels of an Increased Mechanical Resistance Obtained by Microalloying. *Proceedings of the European Congress & Exhibition on PM*, Nice, France, EPMA Ed., 2001, vol. 1, p. 304-309.
26. R.L., ORBAN, On the Microstructure Formation in Sintering by Infiltration of Loose Powders. *Sintering Science and Technology*, The Pennsylvania State University Ed. 2000, p. 253-258.
27. J.D., DWAN, Manufacture of Diamond Impregnated Metal Matrixes. *Materials Science and Technology*, Vol. 14, 2000, p. 896-900.
28. R.L., ORBAN, Mechanical alloying: an overview. *Proceeding of the Second International Conference on Advanced Materials and Technologies "MATEH'98*. UT Press Ed., Cluj-Napoca, Romania, 1998, vol. 2, p. 583-588.
29. T., AIZAWA ET AL., Nano-structured Materials via Bulk Mechanical Alloying. *Scripta Materialia*, No. 44, 2001, p. 1751-1755.
30. R.L. ORBAN, Thermodynamic Approach to Processing of Metal Matrix Composites In-Situ Particulate Reinforced. *Proceedings of the European Congress & Exhibition on PM*, Nice, France, EPMA Ed., 2001, vol. 4, p. 43-48.
31. N. Berolino, SHS Synthesis of FGM for Thermal Barrier Applications. *Advances in Powder Metallurgy and Particulate Materials*. MPIF Ed., USA, 2002, Part 1, p. 168-174.