CHAPTER 8

Combustion Synthesis Melt Casting

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Abstract: The combustion temperature of a highly exothermic reaction can be above the melting point of the end products, which results in the formation of melt-casting products. Combustion synthesis melt casting technique possesses remarkable advantages for the low-cost production of structural and function materials with unique properties and characteristics. In this chapter, some combustion synthesis melt-casting reaction systems developed in recent years, such as refractory compounds, intermetallics, as well as advanced ceramics, are introduced, and the solidification mechanisms are discussed.

INTRODUCTION

The melt-casting process is rather common to fabricate near-net shape materials for metals, alloys, or composites. However, most of the ceramics have not been fabricated using melt-casting. The main obstacles for the application of melt-casting on ceramics lie in three aspects: First, the temperature required for melting ceramic powders is usually too high to be achieved by conventional techniques. Second, the melt-casting is difficult to eliminate pores, which decreases their mechanical properties. Third, the slow cooling rate means long duration at elevated temperature after crystallization of the melt, which leads to coarse grains with poor toughness or low hardness [1-3].

The method of self-propagating high temperature synthesis (SHS) was founded by Merzhanov and coworkers in the 1960s [4]. SHS process evolves large sums of heat and forms a solid product in the chemically active systems, and shows some excellences such as self-sustaining reaction, high purity, high productivity and so on [5-8]. One of the unique SHS process is combustion synthesis melt casting. When the combustion temperature of the combustion reaction is above the melting point of the end products, melt-casting bulk materials can be obtained. The combustion synthesis melt-casting process combine the advantages of SHS and melt casting techniques and develops a convenient and economic approach to obtain dense, near-net shape components, especially ceramics.

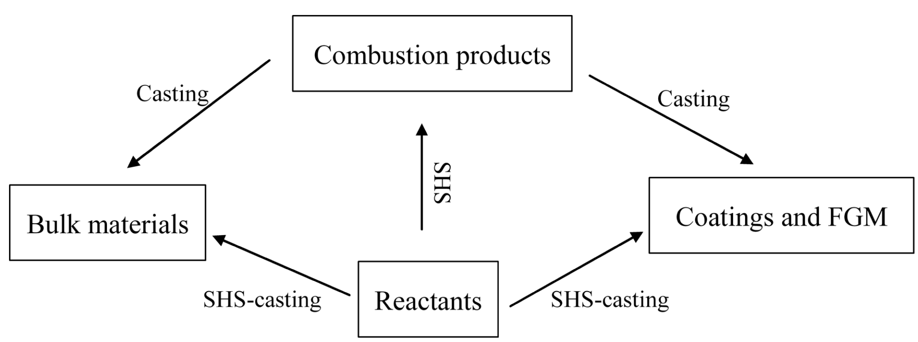

Figure 1: Processing routes in combustion synthesis melt casting technology

As can be seen in Fig. 1, the combustion synthesis melt casting products, including bulk materials, coatings and functionally graded materials (FGMs), can be obtained by combustion synthesis melt casting or by direct SHS. Some of the combustion synthesis melt casting materials and techniques are listed in Tab. 1. It shows most of the interests are fastening on the research of new approaches of syntheses of hard alloys, multifunctional materials, ceramics, and various composites. The attractive direct synthesis consists of centrifugal-casting and pressure-assisted casting.

Table 1: Combustion synthesis melt casting techniques and prepared materials.

types	Compositions	Techniques	Refs
Bulk	Mo_5Si_3	Microwave activated SHS	[9]
	TiB_2-TiC	High-pressure SHS	[10]
	Fe-Cr-Ni/Al_2O_3	Thermite reaction	[11]
	Ni-Ti-C/B_4C/B	SHS-arc melting-suction casting	[12]
	Ti base/TiB	SHS- non-consumable arc-melting	[13]
	Cu-$MoSi_2$	SHS-casting	[14]
	Ti-Si	SHS-casting	[15]
	Zr–Si	SHS-casting	[16]
	Sn-Pb	High-pressure centrifugal infiltration	[17]
	TiC-TiB_2-Me_xO_y	pressure assisted-SHS	[18]
	MgB_2	SHS-casting	[19]
	NiSiCr	Applied gas pressure assisted SHS	[20]
	$MoSi_2$-SiC	Applied gas pressure assisted SHS	[21]
	Ni_3Al	Applied gas pressure assisted SHS	[22]
	FeAl	Field activated pressure assisted synthesis	[23]
	Al_2O_3	SHS-ultrahigh gravity	[24]
	C-C	CS	[25]
	Cu-TiB_2	SHS-quasi-static consolidation	[26]
Coating	TiC/Ni_3Al	SHS-casting	[27]
	IrAl	SHS-casting	[28]
	NiAl	SHS-high concentrated solar energy	[29]
	TiC-TiB_2/Fe	SHS-argon arc cladding	[30]
	NiAl	Centrifugal thermite	[31]
	$Al_2O_3TiO_2TiC$/ AlFe-AlCrFe-NiFe	SHS-centrifugal casting	[32]
	$MoSi_2$-MoS_2	Applied gas pressure assisted SHS	[33]
	Ni_3Al-Cr_7C_3	Applied gas pressure assisted SHS	[34]
	Ni-Cr-TiC	SHS- plasma densification	[35]
	FeCr-TiC	SHS-laser glaze	[36]
	TiC-Al_2O_3	SHS-hot pressing	[37]
FGMs	$MoSi_2$-TiB_2	SHS- vapor deposition	[38]
	Fe-TiC	SHS-centrifugal casting	[39]
	Ti-B	SHS-punching	[40]
	$MoSi_2$-SiC	SHS-hot pressing	[37]
	$MoSi_2$/Al_2O_3	SHS-tape casting	[41]
	Al_2O_3/YAG/YSZ	SHS-high gravity	[42]

In this chapter, some combustion synthesis melt-casting reaction systems developed in recent years, such as refractory compounds, intermetallics, as well as advanced ceramics, are introduced, and the solidification mechanisms are discussed.

COMBUSTION SYNTHESIS MELT CASTING TECHNIQUES

Combustion Synthesis Melt Casting Bulk Materials

Large numbers of the bulk materials have been prepared by combustion synthesis melt casting in recent years. The bulk materials contain metal matrix composites, intermetallics, ceramics, and so on. These combustion synthesis melt casting materials possess unique microstructures and excellent mechanical and chemical properties, such as high strength, oxidation resistance, corrosion resistance and wear resistance, etc.

Starting from powder mixture of MoO_3–CuO–Al–Si system, Cu–$MoSi_2$ composite has been fabricated successfully by the SHS and casting [14]. Four dense ceramic particulate reinforced nickel matrix composites have been fabricated from Ni–Ti–C, Ni–Ti–B, Ni–Ti–B_4C and Ni–Ti–C–B systems, respectively, by SHS reactions, arc melting and suction casting [12].

Compared with traditional casting materials, the combustion synthesis melt casting bulk products show good properties, such as high strength, oxidation resistance, corrosion resistance and wear resistance, etc. Przybylski *et al* [19] have shown that the bulk MgB_2, which has been fabricated by this technique, is a hard, type II superconductor. The superconducting transition of the MgB_2 is very sharp and very sensitive to the *ac* and *dc* magnetic fields. The critical current density is extracted from the absorption susceptibility via a superconducting critical state model. The bulk Ni_3Si intermetallics with 20, 30, 40 wt% Cr have been fabricated by using combustion synthesis melt casting under 7 MPa applied gas pressure [20]. All of the NiSiCr alloys consist of β_1-Ni_3Si, β_3-Ni_3Si and eutectic (β_1-Ni_3Si+ Ni_5Si_2) (Fig. 2). The amount of β_3-Ni_3Si phase decreases as the Cr content decreases. The micro-hardness of the alloys increases with Cr content and the alloy with 30 wt% Cr possesses the highest compressive strength (1.8 GPa). The wear rate of the NiSiCr alloys decreases with Cr content (Fig. 3). It indicates that the optimal properties can be obtained by modifying the compositions of the combustion synthesis.

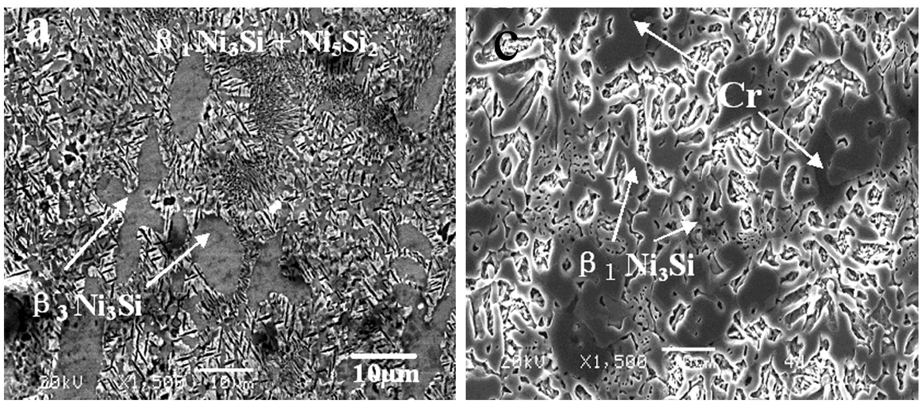

Figure 2: Microstructure of the Ni_3Si alloy with 20, 30, 40 wt% Cr[20]

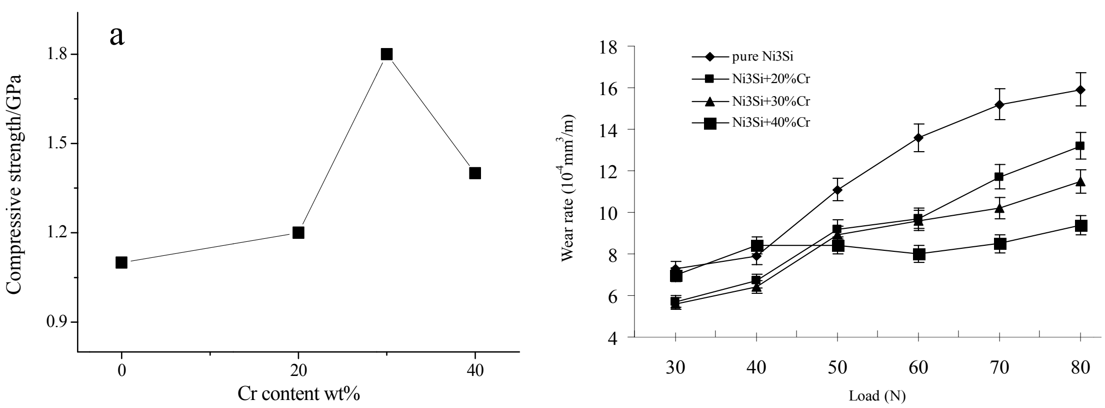

Figure 3: (a) micro-hardness of the NiSiCr alloys, (b) wear rates of the NiSiCr alloys with applied loads at sliding speed of 0.05m/s[20]

La *et al* [21] also studied the relation of the compositions and properties of the bulk $MoSi_2$–SiC composites, which were also prepared under 7 MPa applied gas pressure. The toughnesses of the composites with 10, 15 and 20 wt.% SiC are investigated comparatively. Silicon carbide phase presented in the composites is in the form of large particles or short fibers depending on the amount of SiC (Fig. 4). Cracks exist near interfaces of the large SiC particles and the matrix in the composites with 10 and 15 wt.% SiC, whereas not appears in the composite with 20 wt.% SiC. The higher of the SiC content is, the finer the particles and fibers become, and the more the areas of interface exist. The areas of interface increase with the increasing of the SiC content. Therefore, the toughness of composite increased with the increase of the SiC content(Fig. 5). It proves that the toughness of brittle materials can be improved by second phases strengthen effects [43, 44].

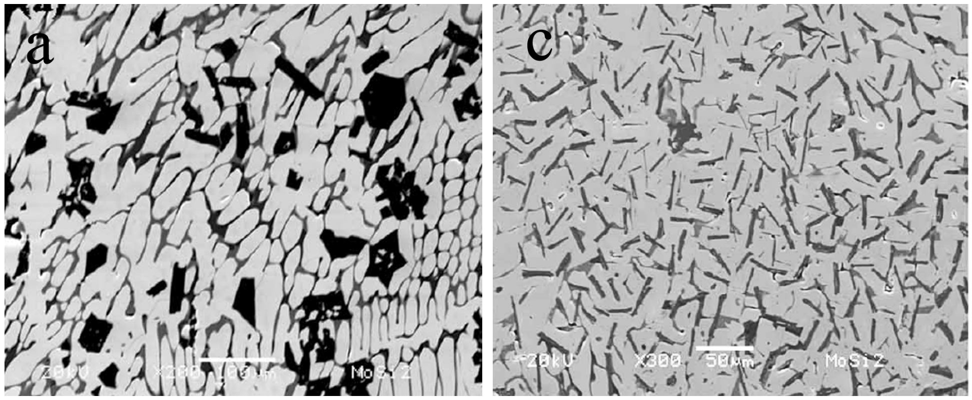

Figure 4: Microstructure of the $MoSi_2$–SiC composites with 10 (a), 15 (b), 20 (c) wt% SiC[21].

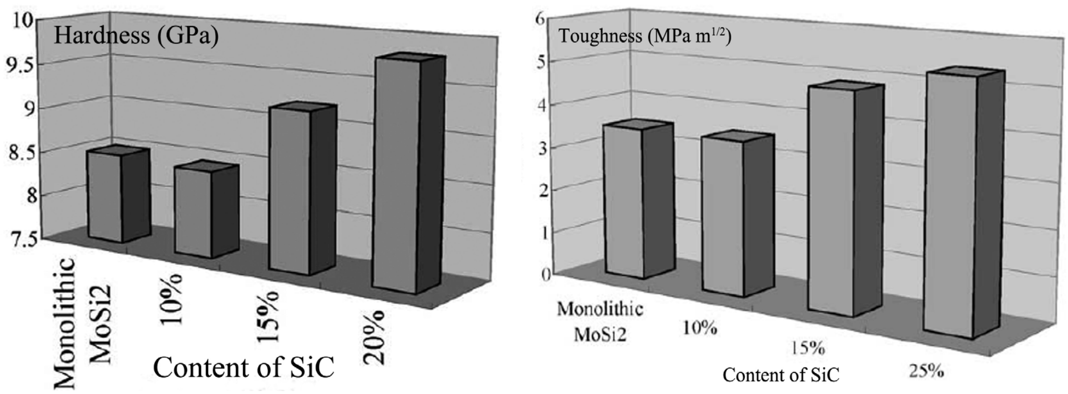

Figure 5: Hardness and toughness of the $MoSi_2$–SiC composites[21]

A major limitation of combustion synthesis melt-casting bulk materials is the high porosity in the materials, which restricts their practical applications. The pores originate from the volume change caused by the different density between reactants and bulk products. On the other hand, evaporation at high reaction temperatures and the pores already present in the "green" reactants sample also result porosity. Recent researches have shown that this limitation can be minimized or even eliminated by subjecting the reactants green body to a consolidating load or by utilizing assistant techniques in the combustion synthesis process, such as adding a proper pressure, high gravity field, and centrifugal force.

A full density TiC–TiB_2 composite was prepared by the combustion synthesis melt-casting in a high-gravity field [10]. The results of Zhao *et al* indicated that the key factor controlling the densification process of TiC–TiB_2 is the liquid phase formed in the products. They found that the reactants consisting of TiC–TiB_2 and oxide impurities are in the full liquid state after combustion synthesis. The lower density oxide liquid rise to the top layer and the product sink to the bottom under the high gravity field (>200g). Moreover, the high gravity also accelerates the escape of gas from product liquid. Hence, the introduction of a high gravity field plays a predominant role in the densification process of the TiC–TiB_2 composites.

The dense Al_2O_3 ceramic was obtained by integrating the combustion synthesis technique with the ultra-high gravity, which originated from the centrifugal force. Pei *et cl* [24] designed the equation $2Al + 3NiO = 3Ni + Al_2O_3$ to obtain superheated melt consisting of molten mixture ($Ni + Al_2O_3$). The melt mixture could be separated successfully by their density difference at the gravitational acceleration magnitude over 200g. However, there are a few pores present in the as- prepared Al_2O_3. The authors point out that the pores may be possible eliminated completely from the molten Al_2O_3 if the *G* value further increases. In fact, a quite dense layer of the α-Al_2O_3 (3.70 g/cm^3) obtained as the gravitational acceleration increases to 800g.

Grain-refined intermetallics, metastable crystalline metals, quasi-crystals, metallic glasses, supersaturated solutions and near-net components can be obtained by precise design the combustion synthesis reaction and proper control the cooling process of the undercooled melts.

In order to further improve the density and properties of the combustion synthesis melt-casting bulk materials, many attempts have been made in the authors' laboratory. The results show that the dense products may be obtained when the highly exothermic reactions are carried on under a higher applied gas pressure as well as rapid cooling subsequently [22]. La *et al* have shown that the density of the bulk W_2C, which being prepared by this technique using the high-exothermal reaction $2WO_3$ +4Al+C=W_2C+2Al_2O_3+3360 kJ/mol, is 98% of the theoretical value. Moreover, the nanocrystalline microstructure of the W_2C is obtained (Fig. 6). The nanocrystalline W_2C shows the micro-hardness (7.5GPa) and toughness (13.5 MPam$^{1/2}$). Although the micro-hardness is lower than that of the W_2C with a microcrystalline structure, the toughness is improved markedly. A nanocrystalline Fe_3Al intermetallics also is produced by the aluminothermic reaction of $3Fe_2O_3 + 8Al = 2Fe_3Al + 3Al_2O_3$ [45]. The nanocrystalline Fe_3Al intermetallics consists of roughly equiaxed grains with uniform grain size of 15-25 nm and with random orientation (Fig. 7). The bending strength of the Fe_3Al product is about 1170 MPa and the yield strength is 1050 MPa, Which is much higher than that of the reported yielding strength of 500 MPa of Fe_3Al with a microcrystalline structure, and the Fe_3Al product exhibits excellent ductility (~70%) in compressive tests. Using this technique, lots of nanostructured materials had been processed, such as Ni_3Al, Fe_3Al, NiAlCr, AlCuFe, FeC, FeSi, FeB, FeCu, CuAl, CuAlFe [22, 45-50].

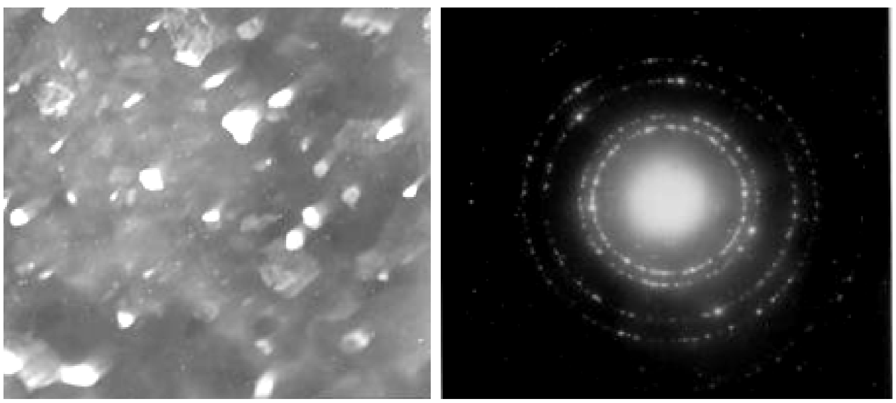

Figure 6: Dark field Transmission Electron Microscope (TEM) micrograph and corresponding Selected Area Electron Diffraction (SAED) pattern of the nanocrystalline W_2C[22].

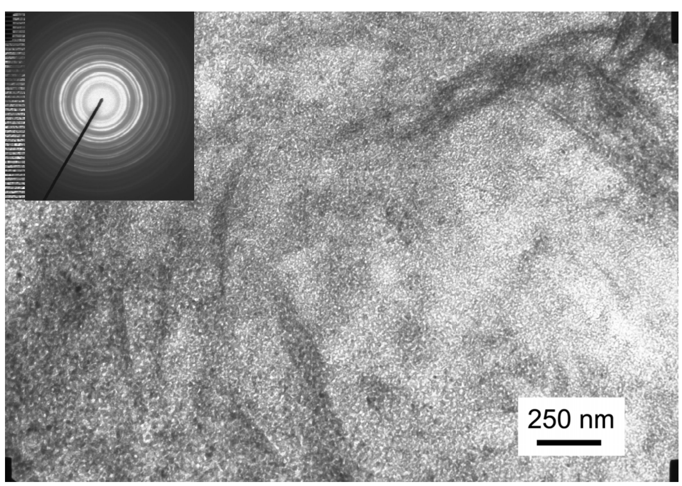

Figure 7: Bright-field TEM image and corresponding SAED pattern (inset) of the nanocrystalline Fe_3Al intermetallics[45].

It indicates that the combustion synthesis melt-casting method is a very effective one for tailoring nanostructured and other metastable metals, quasi-crystals, metallic glasses, supersaturated solutions materials. Details of these parts will be expounded in chapter 10.

Combustion Synthesis Melt Casting Coatings

There are lots of techniques, including chemical vapor deposition (CVD), electronic deposition, plasma spraying and centrifugal casting, which have been developing to fabricate advanced coatings. The combustion synthesis melt-casting techniques also have been applied to the fabrication of coatings on substrate materials. The combustion synthesis melt-casting coatings can dramatically improve the mechanical and chemical property and especially, the wear resistance of the surface [37, 51].

Ode *et al* [28] reported that IrAl coating on metal substrates can be obtained by the combustion synthesis melt-casting process of the reaction Ir+Al. However, the coating on the metal substrate contains some powders that have not reacted and has poor adhesion strength with the substrate. Although the combustion synthesis reaction can be considered an adiabatic process, there are actually still some heat losses by conduction to the substrate materials and radiation from the coating surface during the reaction. Therefore, in order to maintain the self-sustaining reaction and to improve the adhesion strength, substrate preheating is required. Sierra *et al* [29] studied the heat conduction of NiAl coating on steel. When the Ni+Al reaction is ignited, the combustion heats raise the substrate temperature. If the temperature is high enough, the NiAl coating and the surface layer of the steel substrate are in a molten state, and Ni, Al and Fe diffusion occurs, and high adhesion strength between the NiAl coating and steel can be obtained. It is found that the heat released in the reaction depends on the mass of the Ni+Al reactants, for example, a sample with 1.7 g NiAl releases enough heat and obtain a satisfing coating, but samples with 0.6 g NiAl or less are not enough to achieve a fine coating.

Other researchers hope to apply highly exothermic reaction to resolve this problem. By following the two equations:

$$MoO_3+2Al+2Si=MoSi_2+Al_2O_3+1051.7(\text{kJ mol}^{-1})$$

$$MoO_3+2Al+2S= MoS_2+Al_2O_3+ 1170.2\ (\text{kJ mol}^{-1})$$

The dense $MoSi_2$-MoS_2 (6-12 wt.%) composite coatings on a commercial carbon steel 1045 substrate have been prepared by combustion synthesis melt-casting route at 400 ^{o}C and 5 MPa applied gas pressure [33]. The coatings have a dense microstructure and are fully bonded with the substrate through metallurgical bonding. The interface morphology of the coatings with 6 and 12 wt.% MoS_2 are shown in Fig. 8, where the section at the left refers to the coating; and the right side to the substrate. There is an interface phases region in which are composed of Fe from substrate and ceramic phase. No cracks or pores near the interface region.

The other research about W_2C-10vol.%W coating on the 45# steel also indicated that the effective diffusion of the interface atoms can improve the bonding strength [22]. The W_2C-W (10vol.%) coating is composed of α-W_2C, $(W_2C)_{120}$ and W phases (designated as white zone, gray band and black point, respectively, Fig. 9a). The Energy Dispersive Spectroscopy (EDS) results show that W in coating and Fe in the substrate diffused sufficiently (Fig. 9b), which results in good adherence of the coating and the substrate.

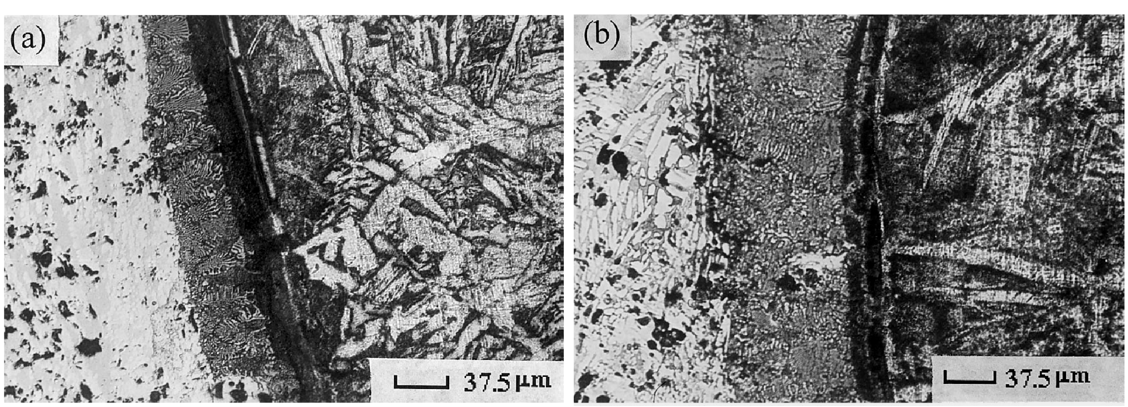

Figure 8: Interface Optical Microscope (OM) micrographs of the $MoSi_2$-MoS_2 coatings with 6 wt.% MoS_2 (a) and 12 wt.% MoS_2 (b) [33].

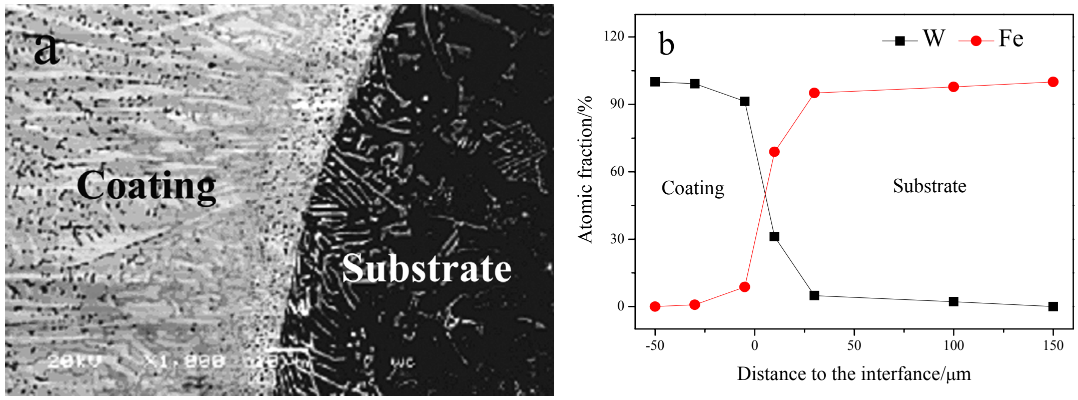

Figure 9: Interface OM micrographs of the W_2C-10vol.%W coating (a) and distribution of the Fe, W elements in the interface (b) [22].

Centrifugal-thermite process is considered to obtain dense, well-bonded castings. Shaped casting coatings of ceramic composites and intermetallic composites can be produced by *in situ* synthesis and densification of ceramic lining to metal substrates under the influence of gravity. Many investigations have examined the use of centrifugal-thermite reactions to coat steel pipes with ceramic layers in recent years. Du *et al* [52] studied the effects of additives such as SiO_2, CrO_3, $Na_2B_4O_7$ and ZrO_2 on the densification degree of the ceramic lining copper pipe, which was produced by centrifugal-thermite process. The results show that the densification degree of the ceramic layer can be improved by adding SiO_2 and CrO_3 in the thermite. The bonding strength is increased through the addition of a suitable amount of $Na_2B_4O_7$. But the highest fracture toughness of the ceramic layer is obtained at 7 wt.% of ZrO_2 in thermite.

However, there are still now a lot of problems present in the melt casting coatings, for example, the structural disadvantages at the metal/ceramic interface in ceramics lining pipes fabricated by centrifugal-thermit process and the poor bonding strength of bend pipe have limited their application. And also these problems motivate further studies on combustion synthesis melt-casting coatings.

Combustion Synthesis Melt-Casting Fgms

Direct bonding of ceramics and metals can generate thermal stresses in practical high-temperature applications because of mismatched thermal capacity. The thermal stress causes flaw or debonding at interfaces. The functionally graded materials (FGM) can effectively solve this problem. A well-known example is applied to create the fuselage exterior and engine materials for space planes which would take off like airplanes, cruise in the atmosphere subject to severe frictional heating from the airflow. To fulfill this requirement the composition and microstructure are varied throughout the structure and this yields a property gradient within the combined materials. The FGM is also introduced to the combustion synthesis melt-casting techniques for improving bond strength of the interface. The in-situ reinforced Fe–TiC (Fe–TiC) functionally graded materials can obtain by combustion synthesis melt-casting followed centrifugal casting in a single step [39].

A rapid and simple way of producing Al_2O_3/YAG/YSZ ternary eutectics FGM by combustion synthesis melt-casting was investigated [42]. The combustion reactions between $Al/Fe_2O_3/Y_2O_3/ZrO_2$ led to the formation of molten mixtures consisting of Al_2O_3/YAG/YSZ. The formation, separation and densification of molten compounds consisting of Al_2O_3/YAG/YSZ and iron are realized under an ultra-high-gravity field in a short time. The as-solidified ceramic ingot sank into the iron melt, where an instantaneous isostatic pressure about 2 MPa was exerted on the around of the ceramic ingot, which results in an enhanced degree of densification.

Combining the graded composition and proper pressure, the adherence of the interface was further enhanced. $MoSi_2/Al_2O_3$ functionally graded materials (FGMs) with alumina contents varying from 20 to 80 mol% have been fabricated using a combination of tape casting and pressures-assistant high-temperature combustion synthesis [41]. The applied pressure, between 0.9 and 3.4 MPa, significantly increased the adherence between the layers and the density of the FGM. When a 1.4 MPa pressure was applied before the ignition and maintained during the combustion synthesis and cooling period, the adherence between the different layers became satisfactory but a large amount of porosity still remained. But the porosity decreased significantly when the applied pressure increased. The larger pores completely disappeared when the applied pressure was 3.4 MPa.

APPLICATIONS

Compared with conventional melt casting processing, the advantages of combustion synthesis melting casting are following: No or little external energy is needed; The low boiling point impurities and by-products can be volatilized in the high reaction temperature, which results in higher purity products; The overheated and undercooled melt can led to new or metastable materials, such as nanocrystalline, amorphous or immiscible alloys. The authors studied the economical effectiveness of production of aluminium nitride from the same raw materials using combustion synthesis melting casting, furnace (FS) and plasmochemical (PCS) techniques, the results are given in Tab. 2. It shows that the combustion synthesis melting casting technology superior than the other alternative process by almost all the parameters.

Actually, the combustion synthesis melting casting technique and its products can find the application in lots of areas. Their applications can be classified as follows: electro insulating corrosion-resistant bushings; electrolysis bath protection; crucibles for ferrous metal and amorphous alloys; lining of high-temperature heaters; biological protection; erosion-resistant burner bushings; plasma cutting of metals and alloys; "Pouring" amorphous tapes; sintering fixture for heat-conducting boards; parts of internal combustion engine; metallurgical fixtures; parts for metallurgical industry, black ceramics; ball bearings; parts operating at high temperatures; high temperature intermetallic compounds and so on [5, 37].

Table 2: Industrial parameters of SHS, FS, PCS synthesis process [53].

Parameter	SHS	FS	PCS
Consumption of the raw materials			
Aluminum/kg	o.7	0.9	1.5
Nitrogen/m^3 (kg^{-1})	0.9	1.65	12.3
Consumption of electric energy/kWh kg^{-1}	0.5	31	150
Labor consumptions (rel. unit)	1	1.4	3.5
Number of technological stage	8	18	5
Effectiveness (kg h^{-1})	4.0	1.0	0.75
Time of technological stage/h	0.6	2.5	0.5
Net cost power (rel. unit)	1	2	4

Some of the combustion synthesis melting casting components from the product or mixture of products of the thermit reaction are give in Fig.10a [54]. Fig. 10b shows some machine parts made of black ceramics, which the strength remains constant up to 1500 ^{o}C [5]. Large-sized pipes with internal wear-resistant coating for transportation of abrasive media and pipelines have been scaled up to yield in China (Fig. 10c) [55]

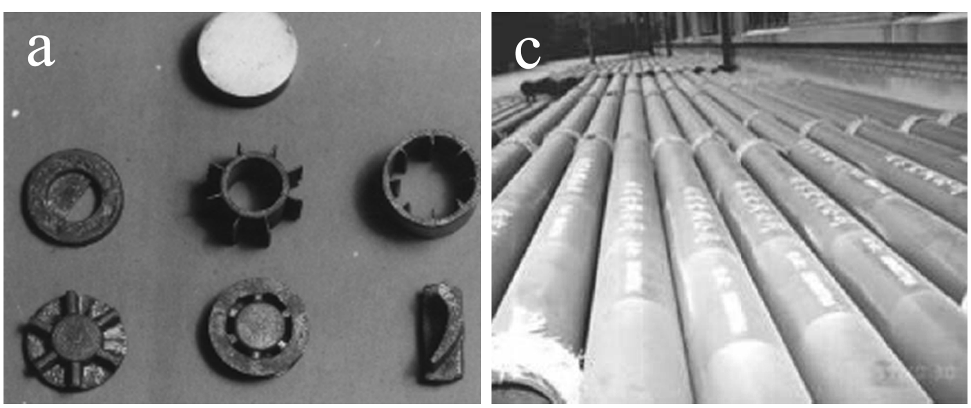

Figure 10: Combustion synthesis melt casting products (a) Shaped Casting of Nickel Alluminide[54], (b) ceramic items[5], (c) large-scale ceramic-lined steel pipes[55]

SUMMARY

The combustion synthesis melt casting has considerable potential in the production of bulk materials and coatings or FGMs. The relative simplicity of the combustion synthesis melt casting technology provides an advantage in terms of both energy and cost saving. The development of the combustion synthesis melt casting technique will run into the highway in the near future.

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