Role of Ceramics in a
Self-sustaining Environment
R. Pampuch and K. Haberko (Editors)

ENERGY-SAVING TECHNOLOGIES

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Energy-saving technologies based on self-propagating high-temperature synthesis (SHS) are considered. Discussed are the classes of chemical reactions and synthesized products as well as the most popular technological types. The advantages and drawbacks of SHS as a challenge to conventional technologies were formulated. Fundamentals and concrete examples of energy saving due to implementation of SHS technologies were considered in brief. Research directories that characterize SHS as a technology-intensive process were enumerated. The prospects for energy saving in SHS technologies were outlined.

INTRODUCTION

Nowadays development of new efficient energy-saving technologies is of great importance for modern civilization. In this context, the following two energy-related principles (electrical and chemical) of high-temperature processes of chemical technology are worth mentioning.

The first one is conventional. Produced electric power is then used to obtain high temperature in reactors. Within this scheme, high temperature is attained twice: upon fuel combustion at an electric power station and then in a reactor. In this case, losses are unavoidable. But this is a universal principle applicable to any kind of technological processes.

The applicability of the second principle is confined to the class of exothermic reactions. In this case, high temperature is attained due to internal release of chemical energy rather than external heat supply, heat losses being minimal. For this reason, chemical technologies utilizing the second principle are termed energy-saving.
It is clear that the higher is the processing temperature, the more efficient is the chemical-energy approach and the higher is energy saving.

Combustion is the best way to utilize chemical energy for self-heating of substance for technological purposes. High rate of chemical reaction, no need for preheating, and simple facilities – all this makes the process highly convenient. It was termed "technological combustion".[1]

The following are some widely known processes of technological combustion in metallurgy and chemical industry[2] :production of pig iron in blast furnaces, of ferroalloys and alloying agents by out-of-furnace alumothermy, of process gases and carbon black upon incomplete combustion of hydrocarbons, of inorganic acids by technological combustion of gases.

A method that allows us to use technological combustion for the synthesis of ceramics and ceramic materials was developed in 1967 at the Research Center of the USSR Academy of Sciences in Chernogolovka. It was termed self-propagating high-temperature synthesis (SHS) [3-4] (see also review papers [5-12]).

SHS is a special kind of combustion (in the mixture of powder reagents) that yields solid products with valuable properties. The photo and schematic of SHS process are presented in Fig. 1.

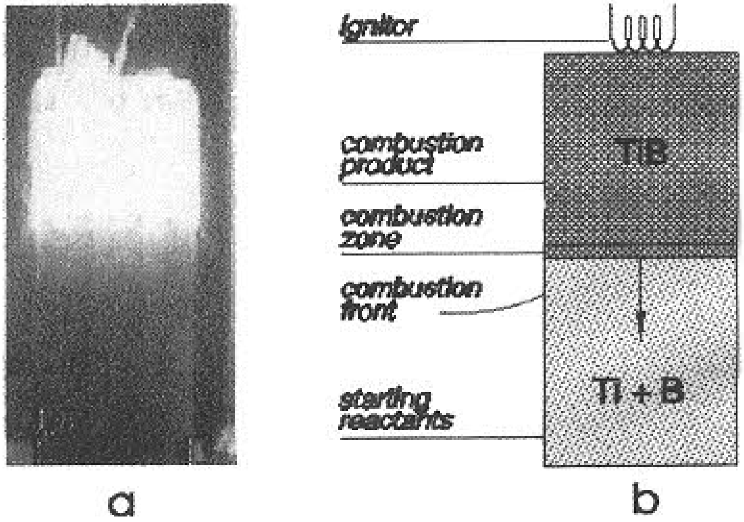

Fig. 1. Photo (a) and Shematic (b) of SHS Process.

SHS process involves the following stages typical of any kind of SHS processes: ignition of a starting sample, front propagation, and sample cooling after combustion. Typical parameters of SHS process are presented in Table 1. High burning velocities in the condensed phase, high combustion temperatures, and high heating rates allow us to regard SHS as an extreme chemical process.

This paper deals with the application of SHS to manufacturing ceramics. An incomplete list of ceramics that have already been prepared by SHS is presented in Table 2. These are non-oxide refractory compounds (both metallic and nonmetallic), complex (mixed) oxides, and other ceramic materials of different composition and destination.

CHEMICAL ASPECTS

The chemistry of SHS process differs from that of conventional ceramic processing.[6] Necessary condition for SHS is the availability of exothermic reactions or stages that would sustain the propagation of combustion wave in the system. A general scheme of SHS process can be represented in the form:

$$\sum_{i=1}^{m} R_i(s,l,g) \rightarrow \sum_{j=1}^{n} P_j(s)+Q,$$

where R_i is a reactant, P_j is a product, Q is the heat release. s, l, g stand for solid, liquid, and gas, respectively.

Table 1. Typical Parameters of SHS Process

Particle size, r	0.1 - 50 μm
Relative sample density, Δ	from the bulk one to $\Delta = 0.6$
Sample diameter, d	
laboratory	0.5 - 1.5 cm
industrial-scale production	0.2 - 0.4 m
Burning velocity, U	0.1 - 10 cm/s
Peak temperature, T_m	1500-3500 K
Heating rate in the wave, W	10^3 - 10^5 K/s
Heat release in reaction, Q	200 - 1500 cal/g
Ignition power, q	10-200 cal/cm^2-s
Ignition delay time, t_{ign}	0.1 - 1.0 s

As a rule, SHS systems contain a combustible and an oxidizer. Metals and nonmetallic elements normally act as combustibles, while boron, carbon, silicon, nitrogen, oxygen, and some their compounds are used as oxidizers. The following are examples of chemical reactions in ceramic SHS:

(i) synthesis from elements (solid flames)

$Zr + 2B \rightarrow ZrB_2$
$Ti + C \rightarrow TiC$
$Mo + 2Si \rightarrow MoSi_2$

(ii) synthesis from elements (infiltration flames)

$Ti + 1/2\ N_2 \rightarrow TiN$
$3Si + 2N_2 \rightarrow Si_3N_4$

(iii) synthesis with a reducing stage (metallothermic SHS)

$TiO_2 + B_2O_3 + 5Mg \rightarrow TiB_2 + 5MgO$
$B_2O_3 + 3Mg + N_2 \rightarrow 2BN + 3MgO$
$3Cr_2O_3 + 6Al + 4C \rightarrow 2Cr_3C_2 + 3Al_2O_3$

(vi) synthesis of complex oxides

$3Cu + 2BaO_2 + 1/2Y_2O_3 \xrightarrow{O_2} YBa_2Cu_3O_{7-x}$

Note that the presence of oxygen is unnecessary for combustion. In SHS, the examples of oxygen-free combustion are numerous. One of most prominent is the synthesis of hydrides, where hydrogen, the well-known combustible, acts as oxidizer.

Discussing the chemistry of SHS processes, we considered only the overall reaction schemes. In reality, SHS reactions involve numerous intermediate stages. Their identification is one of objectives of SHS chemistry.

The chemical mechanism of the synthesis of yttrium barium cuprate, the well-known high-*Tc* superconductor, is presented below as an example:[13]

overall reaction scheme

$$3Cu + 2BaO_2 + 1/2Y_2O_3 \xrightarrow{O_2} YBa_2Cu_3O_{7-x}$$

actual reaction mechanism

during preheating and in the zone of main heat release (up to ~ 830°C)

$2Cu + O_2 \rightarrow Cu_2O + 1/2O_2$
$BaO_2 \rightarrow BaO + 1/2O_2$
$(BaO + BaO_2)_{melt} + 2Cu_2O \rightarrow 2BaCu_2O_2$
$(BaO + BaO_2)_{melt} + 2CuO \rightarrow 2BaCuO_2$

in the after-burning zone (Tm ~ 950°C)

$2BaCu_2O_2 + CuO + 1/2Y_2O_3 \rightarrow YBa_2Cu_3O_{6+\delta} + Cu_2O$
$BaCuO_2 + CuO \rightarrow BaCu_2O_2 + 1/2O_2$
$2BaCuO_2 + CuO + 1/2Y_2O_3 \rightarrow YBa_2Cu_3O_{6+\delta}$

in the cooling zone

$YBa_2Cu_3O_{6+\delta}$(tetra) $\rightarrow YBa_2Cu_3O_{7-x}$ (ortho)

This product is extremely capricious. To prepare high-quality product, one has to know the mechanism of SHS reaction in detail.

CERAMIC SHS TECHNOLOGIES

SHS technology has three types of objective. One is conventional chemical synthesis, when only the product composition should meet strict requirements. In this case, a typical objective is preparation of single-phase compounds. Another is direct synthesis of ceramic material with additional requirements to micro- and macrostructure of the product. Here, widespread task is direct synthesis of hard alloys. At last, direct (net-shape) production of items. In this case, the end product should meet strict requirements not only to its composition and structure but also to its shape, size, and service parameters. The example of resolved problem is direct synthesis of functionally-graded armor plates.

It is clear that, on going from the first to the third objective, actual difficulties increase markedly.

To resolve these problems, more than thirty modifications of SHS technology has been developed so far. These can be classified into the following six principal technological types:[9]

TT-1 chemical synthesis,
TT-2 SHS sintering,
TT-3 forced SHS compaction,
TT-4 SHS metallurgy,
TT-5 SHS welding,
TT-6 gas-transport SHS deposition.

The first three are the most applicable to producing ceramics. They are considered in more detail below.

The scheme of simplest SHS production is as follows. It is based on the combustion of powder reactants to obtain intermediate products for further processing. These are cakes, shapeless compacts, and blanks which are used as raw materials for preparing powders. Their comminution is carried out by conventional mechanical disintegration or by chemical means. Three types of SHS powders should be mentioned: single-crystal grains, agglomerates, and composite powders.

Applications of SHS powders are versatile, depending on both their chemical composition and particle size. SHS powders are used for: sintering (<3μm), hot pressing (1-10μm), detonation-driven sputerring (20-40μm), and plasma sputtering (40-100μm).

The most popular SHS powders are TiC, TiN, TiC-TiN, TiB_2, $TiC+TiB_2$, TiH_2, AlN, Si_3N_4, Si_3N_4+SiC, BN, B_4C, $MoSi_2$, NiAl, TiNi, Cr_3C_2, $(TiC-Cr_3C_2)+Ni$, $LiNbO_3$, $Mn_xZn_{1-x}Fe_2O_4$, $SrO \cdot 6Fe_2O_3$, $YBa_2Cu_3O_{7-x}$.

Synthesis is conducted either in air or in special reactors. A universal reactor for various syntheses, an open reactor that allows self-purification of reactants from gasifying impurities during combustion, and a flow reactor for syntheses involving gaseous reactants (nitrogen, hydrogen, etc.) became most widely spread.

SHS sintering is a technology used to perform direct sintering of combustion product during SHS process, yelding desired item. The charge is compacted in the form of an item, and SHS is conducted so that its initial shape and size be retained. Progress was made in the following two directions: (*i*) synthesis of nitride ceramics under high nitrogen pressure in high-pressure gasostats and (*ii*) synthesis of ceramics on the basis of carbides, borides, and silicides in evacuated reactors. However, complete sintering is difficult to achieve under these conditions; and this technological type was applied to prepare items with high or low porosity, on retention of their service parameters. By these methods, the following SHS products were manufactured:

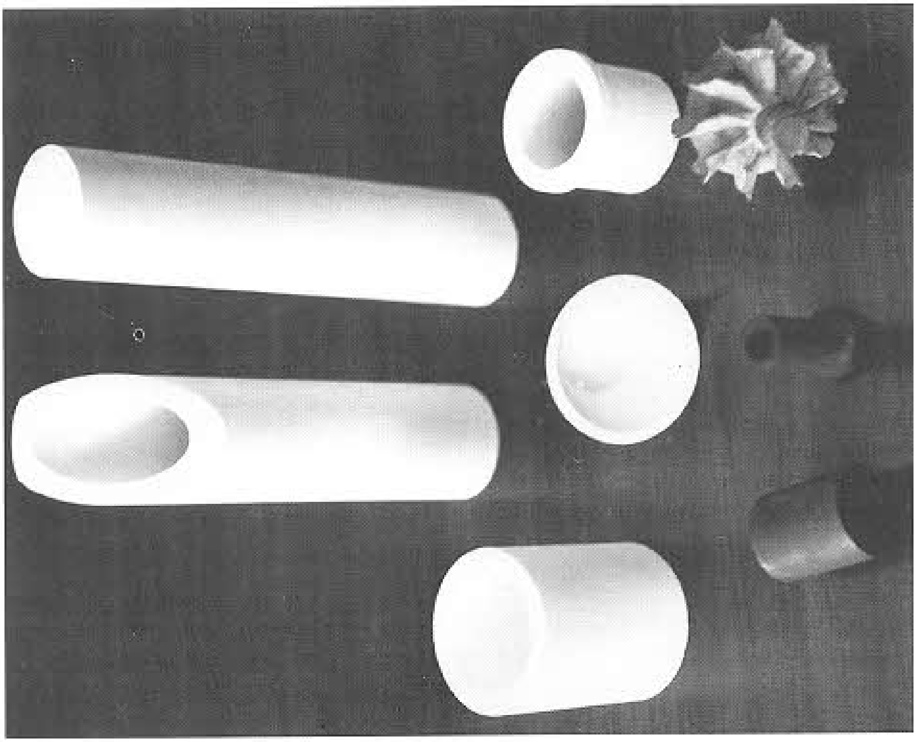

Fig. 2. Photo of "white" and "black" ceramic items

ceramic filters, porous blanks for filters, ceramic insulators, crucibles for metals, blanks of boron nitride, special refractories. These items are exemplified in Fig. 2.

Another very important technological type is the so-called forced SHS compaction. Its essence if fairly simple: still hot combustion product is compacted to its pore-free state and, sometimes, shaped in special utilities. This can be achieved by (1) uniaxial pressing, (2) hot isostatic pressing (HIP), (3) extrusion, (4) rolling, (5) forging, and (6) explosive shock. Best results were obtained (see Fig. 3) in fabricating the STIM hard alloys (cutting inserts, dies, drawing dies, disks, press dies, rolls, etc.).

These and other technological types are applicable according to the overall scheme described below.

The process involves the following three stages:
(*i*) preparation of green mixture followed by quality control. This stage involves drying, weighing, mixing, pelletizing, and shaping;
(*ii*) SHS in air, reactors, gasostates, thermovacuum cameras, press dies, extruders, centrifugal machines, surfacing cameras, etc.;
(*iii*) processing combustion products by crushing, grinding, classification, polishing, cutting, sharpening, etc. of combustion product.

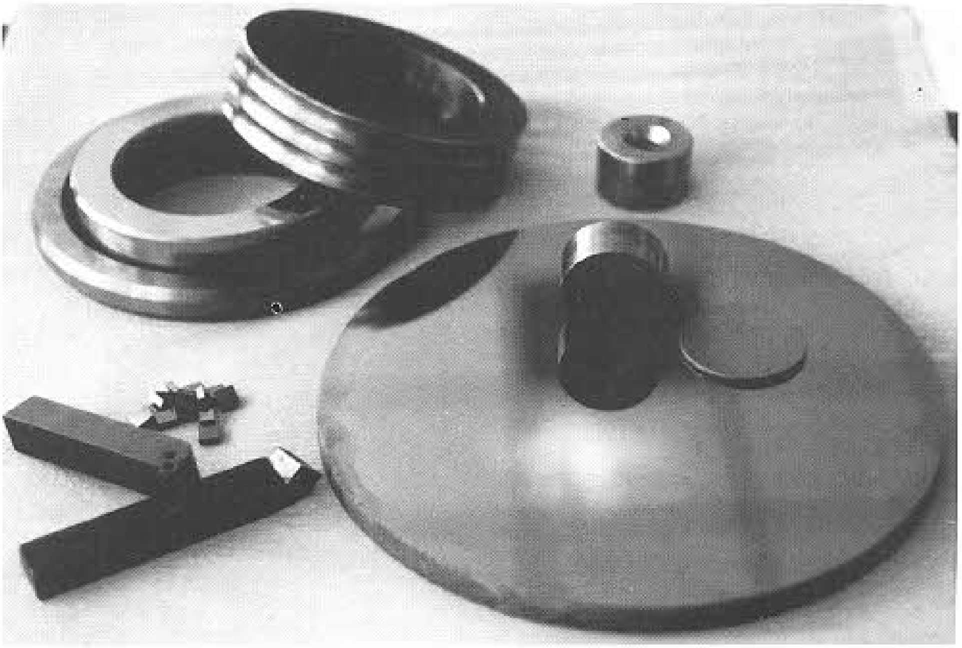

Fig. 3. Photo of STIM hard alloys items.

Table 2. Ceramic SHS Products.

Non-oxide ceramic compounds	Complex oxide ceramic compounds	Ceramic materials
borides	*niobates*	*Silicon Nitride Ceramics*
carbides	*tantalates*	
nitrides	*titanates*	*Boron Nitride Ceramics*
silicides	*cuprates*	
	ferrites	*Hard Alloys*
Metallic compounds (examples)	*etc.*	*(tungsten-free)*
TiC, TiN, TiC-TiN,	examples	*Abrasives*
TiB_2, Ti_5Si_3	$LiNbO_3$	
Non-metallic compounds	$TaNbO_3$	*Refractories*
(examples)	$BaTiO_3$	
	$YBa_2Cu_3O_{7-x}$	
AlN, Si_3N_4, SiC	$(Ni\text{-}Mn)O \cdot Fe_2O_3$	*High-T_c superconductors,*
B_4C, BN *etc.*	$BaO \cdot 6Fe_2O_3$	*etc.*

Table 3. SHS as an Alternative Technology.

Conventional technology	Alternative SHS technology
furnace synthesis, plasmochemical synthesis	simple SHS (TT-1)
sintering, hot pressing, isostatic pressing	SHS sintering (TT-2) SHS densification (TT-3)
cast molding, centrifugal casting	SHS casting (TT-4)
plasma spay and detonation-driven sputtering	gas-transport SHS technology (TT-6) induction SHS surfacing (TT-4)
CVD processes	gas-transport SHS technology (TT-6)
electric-arc and induction surfacing	SHS surfacing, induction SHS surfacing (TT-4)
electric-arc welding	SHS welding (TT-5)

Among them, SHS is a fastest stage. Although stages 1 and 3 are only indirectly related to SHS, their improvement is important for the SHS problem as a whole. Powders (metals, nonmetals, oxides, hydrides, fillers, additives) and gases (N_2, H_2, O_2, inert gases, gaseous compounds) can be used as reactants. This SHS technology is used for production of inorganic compounds (powders) such as borides, nitrides, silicides, hydrides, oxides, chalcogenides, intermetallides, phosphides, etc. Multicomponent materials and items (abrasives, hard alloys, ceramics, refractories, superconductors, items for electrical engineering, surfaced items, joined items, etc.) can be manufactured as well.

SHS as a challenge to conventional processing can be used to solve similar tasks. In Table 3, SHS technology is compared to methods of conventional production methods.

Despite the need for the preparation of exothermic mixtures and some problems with raw materials, SHS technology possesses a number of advantages. The most important are (*i*) high burning velocity, (*ii*) lower cost of chemical energy, (*iii*) rapid layer-to-layer self-heating (instead of slow heating through the surface), and (*vi*) low number of processing steps (down to one-step production).

EFFICIENCY OF SHS PROCESS

Efficiency of SHS process stems from some physical features of the process.[14-15] For instance, high combustion temperature ensures complete chemical conversion, self-purification from some impurities, and elevated rate of homogenization. As a results, this leads to high quality of end products. High burning velocity ensures high productivity. Internal (chemical) heat release ensures saving of resources and, accordingly, lower production cost. The overall efficiency of SHS originates from both lower production cost and enhanced service parameters of end products.

The technical level of SHS products is sufficient for resolving complicate technical problems. Some SHS products have no commercially available analogs (Table 4).

Table 4. Technical Level of SHS Products Examples.
(in Comparison with Commercially Available).

1. Powders AlN, α-Si_3N_4BN	*higher nitrogen content* *(completness of nitriding)* *lower content of impurity oxygen*
2. Composite powders TiC+ αCr_3C_2+ βNi	*no prototype*
3. TiC - based abrasive pastes	*grinding and polishing simultaneously, higher labor productivity*
4. Cutting inserts STIM-5	*higher wear resistance during cutting*
5. Ceramic insulators for the furnaces used in oriented crystallization	*higher service life*
6. Large-scale rolls for copper rolling	*no porotype*

The explanation for higher quality of SHS products is as follows: SHS is a multiparametric process, it can by controlled by a large number of parameters. Therefore, when the optimal combination of parameters is found, high quality of product is guaranteed.

SHS efficiency in the production of aluminum nitride powder is exemplified in Tables 5 and 6. In the former, the characteristics of SHS powders are compared with those of furnace-produced by leading manufacturers. SHS powders are superior over their furnace analogs in the degree of nitriding and oxygen contamination.

The characteristics of three different processing techniques: SHS, furnace, and plasmochemical–are compared in Table 6. The use was made of the data of pilot-scale production in the former Soviet Union. SHS products are superior over their analogs almost in all aspects. Difference in energy consumption and production cost is most impressive.

Table. 5. Characterization of SHS Ceramic Powders (for AlN as an example).

Content of (wt %)	SHS, ISMAN (high quality)	Furnace synthesis, trade companies
Nitrogen (N)	33.9	33.0-33.4
Impurity oxygen (O)	0.3	1.0-2.0
Specific surface m^2/g	2.0-20.0	1.0-8.0

Table 6. Characteristics of SHS, FS, and PCS Techniques.

Processl parameters	SHS	furnace synthesis (FS)	plasmochemical synthesis (PCS)
Raw material consumption			
aluminum, kg/kg	0.7	0.9	1.5
nitrogen, m^3/kg	0.9	1.65	12.3
Electric energy requirements,	3.3	31	150
Labour input, arb.units	1	1.4	3.4
Number of processing steps	8	18	5
Synthesis installation productivity, kg/hr	4.0	1.0	0.75
Powder cost, arb.units	1	2	4

Note. The data refer to pilot-scale production in the former USSR

Considering energy-saving technologies, we should pay a little bit more attention to actual figures characterizing energy saving. The fact that SHS has no need for external energy supply is evident. Moreover, SHS is accompanied by energy release. In reality, energy is spent not only for synthesis but also for some auxiliary operations, and this should be kept in mind. Since different technologies require different raw materials, we should take into account energy consumption for preparation of these raw materials from the same starting products. Here, decisive arguments must come from calculations rather than from logic deductions.

In Table 7, the calculated energy saving upon the substitution of SHS for furnace production of powder titanium carbide is presented. In these calculations, we used the data obtained from one

Table 7. Calculation of Energy Saving Upon Substitution of SHS for Furnace Synthesis (for TiC as an example).

	Furnace synthesis	SHS-1	SHS-2
Chemical reaction	$TiO_2+3C \rightarrow TiC+2CO\uparrow$	$Ti+C \rightarrow TiC$	$TiO_2+2Mg+C \rightarrow TiC+2MgO$
Energy consumption per 1 kg powder, kWh/kg	~ 35	~ 2	~ 3
	Energy consumption per 1 kg powder	Ti ~ 11 kWh/kg Mg ~ 16 kWh/kg	
Total energy saving, including energy consumption for manufacturing metal powder *(but with no regart to energy release in reaction)*			
		SHS-1 ~ 24 kWh/kg	SHS-2 ~ 19 kWh/kg

Table 8. Calculation of Energy Saving (Data of the Plant Producing Nickel-Zinc Ferrite).

	Furnace process	SHS
Chemical reaction	$Fe_2O_3+0.73MnO+0.27ZnO \rightarrow Mn_{0.73}Zn_{0.27}Fe_2O_4$	$0.8Fe+0.6Fe_2O_3+0.6O_2+0.73MnO+0.27ZnO \rightarrow Mn_{0.73}Zn_{0.27}Fe_2O_4$
Energy consumption per 1 kg ferrite powder		
synthesis	12 kW·h/kg	0.04 kW·h/kg
processing	1 kW·h/kg	1.2 kW·h/kg
Energy saving at the plant		11.76 kW·h/kg
Energy consumption per 1 kg Fe		1 kW·h/kg
Total saving, including preparation of Fe powder		11.57 kW·h/kg

of the plants producing hard alloys in the former Soviet Union, while the data on SHS production were taken from the Institute of Structural Macrokinetics, Russian Academy of Sciences. Energy consumption in the production of titanium and magnesium powders was taken from reported data.

In Tab. 8, similar calculated data for energy consumption at one of Russian plants that has implemented SHS-production of ferrites are presented. These data were taken from economic design performed at the plant. In this case, energy saving is also significant.

FUNDAMENTALS OF SHS

SHS production is technology-intensive. It can be controlled only with great understanding of combustion in the system, the mechanism of composition and structure formation in end product, and the means of their control. For this reason, basic research of SHS processes and their products developed extensively. The following are research directions that made fundamentals of SHS:

(i) combustion theory and experimental diagnostics for controlling SHS processes;
nonlinear dynamics for selecting combustion modes;
(ii) thermodynamics of combustion for prediction and selection processing conditions;
(iii) kinetics of high-temperature reactions for understanding the heat release modes;
(iv) high-temperature thermophysics for understanding heat-transfer processes;
(v) combustion chemistry and structural macrokinetics for controlling the quality of SHS product: composition, structure, and properties;
(vi) material science for characterization of end SHS product.

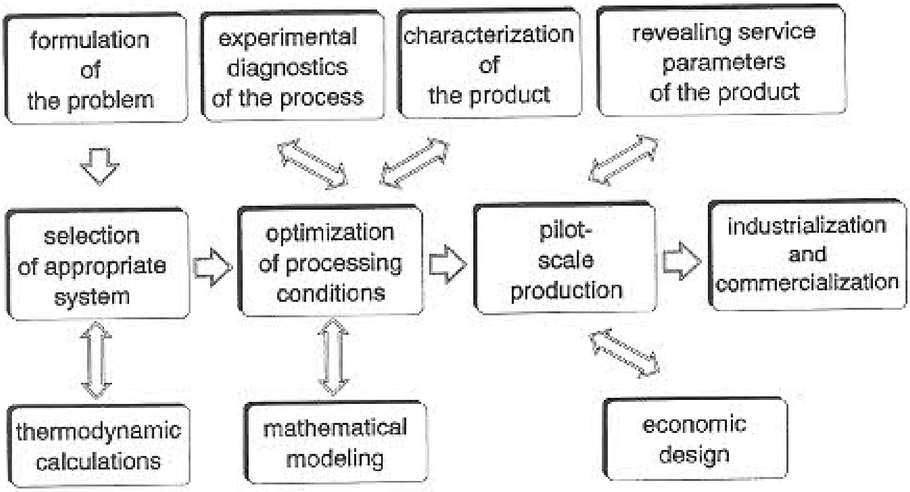

Fig. 4. Chart of Research Work.

Progress in this research allowed formulating the efficient and substantiated means for controlling the burning velocity, combustion temperature, degree of conversion, as well as the composition and structure of combustion products [16-19].

Let us consider only one example how the combustion temperature–most important parameter of SHS process–can be controlled. For well-organized SHS, its maximum temperature (T_m) should be within the range (T'_m $-T''_m$). When $T_m<T'_m$, combustion extincts, SHS does not occur. Otherwise, when $T_m>T''_m$, product quality is unsatisfactory (dissociation, grain growth,

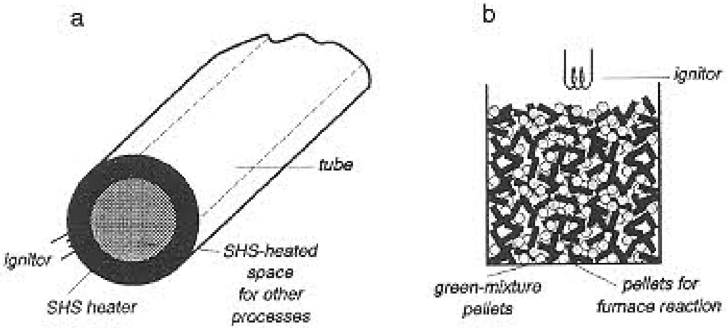

Fig. 5 Two Possible Methods of Released Energy Utilization, (a) SHS-Furnace Process. (b) SHS and SHS-Heating Mixed Process.

macroinhomogenity). An increase in temperature is achieved by external heat supply. The best way to reduce the combustion temperature is a dilution of charge with some amount of end product.

Extensive research and development of SHS allowed us to formulate an organization chart of research work from an original idea to industrial application. It is shown in Fig. 4. Intermediate studies ensure the fidelity of resultant solution.

FUTURE OF SHS AS ENERGY-SAVING TECHNOLOGY

The principal objective of SHS as an energy-saving technology is the development of technologies with the utilization of energy released in chemical reactions. Here the objective is twofold–joint production of materials and energy.
Fig. 5 shows two possible modifications:

(a) SHS-furnace process,
(b) SHS and SHS-heating mixed processes.

The latter is essentially a thermally coupled SHS process. In this case, direct utilization of released heat is possible.

The essence of the idea of SHS electric power station is the following. The heat released during SHS is used not for direct heating but for the conversion to electric power by means of thermogenerator of electric power.

The first variant is more efficient for periodic SHS processes while the second one for continuous SHS.

CONCLUSIONS

The social significance of SHS is as follows: (*i*) saving of resources (energy, raw materials, and labor), (*ii*) development of new high-quality materials and products for application in engineering, technology, and industry, (*iii*) stimulation of new research in combustion theory, general and structural macrokinetics, and in materials science. Energy-saving SHS technologies are now fast developing, and we can expect for new interesting results in the future.

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