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J. W. McCauley

Army Research Laboratory, Aberdeen Proving Grounds, MD, USA

J. A. Puszynski

South Dakota School of Mines and Technology, 501 East Saint Joseph Street, Rapid City, SD, 57701 USA

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Historical Perspective and Contribution of US Researchers into the Field of Self-Propagating High-Temperature Synthesis (SHS)/Combustion Synthesis (CS): Personal Reflections*

J. W. McCauley^a and J. A. Puszynski^b

^a Army Research Laboratory, Aberdeen Proving Grounds, MD, USA

^b South Dakota School of Mines and Technology, 501 East Saint Joseph Street, Rapid City, SD, 57701 USA

e-mail: Jan.Puszynski@sdsmt.edu

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INTRODUCTION

In 1967, Merzhanov, Skhiro, and Borovinskaya published the first comprehensive paper describing self-sustaining character of reactions in a condensed phase, which could be utilized for synthesis of many ceramic and intermetallic materials [1]. In this paper, the authors demonstrated the principle of the so called “solid flame” using reactions between transition metals and boron, carbon or nitrogen. The world-wide combustion synthesis community considers this comprehensive paper and subsequent integrated experimental and theoretical research effort conducted in the former Soviet Union as the beginning of a new approach and method of synthesizing advanced high temperature materials. The main research was conducted by many Russian scientists at the Branch of Russian Academy of Sciences in Chernogolovka under the leadership of Professors Merzhanov and Borovinskaya [2–11].

During that period of our history, free exchange of information among scientists from different countries was very limited due to the cold war. The main source of information on research discoveries and accomplishments of Russian scientists available to US and other researchers was through publications in Russian journals or their translated versions. Such as *Combustion, Explosion, and Shock Waves*, *Doklady Academy Nauk SSSR*, *Soviet Powder Metallurgy of Metals and Ceramics*, *Inorganic Materials*, and *Doklady Physical Chemistry* were the most searched journals in the area of combustion synthesis. In the early 90s, a new *International Journal of Self-Propagating High-Temperature*

Synthesis was created and it is published quarterly since its inception.

Self-propagating high-temperature synthesis (SHS) also called combustion synthesis (CS) is the exothermic process in which the reaction between two or more solid reactants or gas and condensed reactants takes place in a self-sustaining regime leading to the formation of solid products of a higher value [12–14]. During the past forty years, hundreds of different compounds, including, nitrides, borides, carbides, silicides, sulfides, phosphides, hydrides, and oxides of many elements as well as intermetallics, composites, nonstoichiometric compounds, and solid solutions were successfully synthesized by this method [12–18]. Some materials have been successfully scaled-up and produced by the industry. To this group of materials among others belong: carbides of titanium, zirconium, tungsten, tantalum, boron and silicon, titanium diboride, molybdenum disilicide, aluminum nitride, silicon nitride, nickel aluminides, titanium nickelide, zirconium aluminides, and a number of composites (e.g. TiC–TiB₂ and SiC–Si₃N₄) or solid solutions such as SIALONs and aluminum oxynitride (ALON).

REVIEW OF EARLY RESEARCH IN USA AND WESTERN COUNTRIES

A historical perspective on research in the area of exothermic reactions occurring in a self-sustaining regime was well documented by Hlavacek [19] and McCauley [16]. In the United States, the first reported research utilizing self-sustaining character of condensed-phase reactions was conducted by Walton and Poulos [20] in the mid and late 1950s. These authors explored thermite reactions to make refractory coatings. Mixtures of aluminum and/or magnesium with

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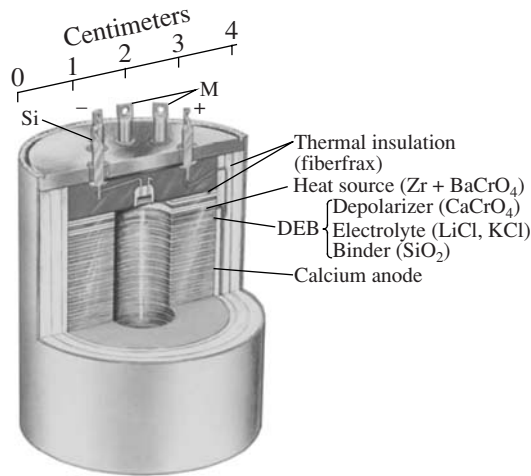


Fig. 1. Use of zirconium in thermal batteries [16].

oxides of iron, cobalt, and vanadium were used to produce different cermet. The authors also explored the combustion synthesis of silicides, borides, and carbides. The use of beryllium as a reducing agent and reduction of uranium oxide were discussed. Several other researchers made attempts to synthesize other materials like aluminum phosphide by direct reaction between red phosphorous and aluminum powders [21], tantalum metal by reduction of K_2TaF_7 with sodium [22], and the formation of molybdenum disilicide by direct reaction between molybdenum and silicon powders [23]. In 1964, Krapf [24] patented the chemical hot press in which a mixture of reactive powders was heated in a die by passing an electric current. After initiation of exothermic reaction, the product was pressed by a uniaxial force. The concept of pressing hot products generated in strongly exothermic reaction was also described in 1967 by Stringer and Williams [25]. According to these authors, reaction pressing can be applied to intermetallic and metal-metalloid compounds generated by fast evolution of energy due to a chemical reaction between reactant powders. They claimed that the exothermic effect of reaction in many cases is sufficient to form plastic product mass which can be quickly formed to different shapes. The authors emphasized the use of aluminides, berrilides, titanides, zirconides, and borides. In 1968, McKenna [26] patented a process of preparing tungsten monocarbide utilizing exothermic effect generated during the reaction between elemental powders. In 1973, Hardt and Phung [27] published a very important paper on propagation of gasless reactions in solids, which further alerted US scientists about importance of the combustion synthesis.

- $Zr + (\text{Air}) \rightarrow ZrO_2$
- $Zr + BaCrO_4 + (\text{Air})$
 - $3Zr + 4BaCrO_4 \rightarrow 3ZrO_2 + 4BaO + Cr_2O_3$
 - $3ZrO_2 + 3BaO \rightarrow 3BaZrO_3$
 - $3Zr + 2BaCrO_4 \rightarrow 3ZrO_2 + 2BaO + 2Cr$
 - $3ZrO_2 + 2BaO \rightarrow 2BaZrO_3$

Heating of reaction:

15–50 wt% Zr 273–502 cal/g
 85–50 wt% BaCrO₄

502 cal/g for ~ 60 wt% BaCrO₄

COMBUSTION SYNTHESIS RESEARCH IN USA AFTER 1980

After sporadic activities in Western World in the 50s and 60s, a more significant research effort was made in the United States starting in early 1980s. In 1982, McCauley et al. [28, 29] and Holt and Kingman [36] published new results in the area of combustion synthesis, which generated interest at several universities and US government laboratories. The review paper on the SHS activities in Soviet Union written by Crider [37] also stimulated a renewed interest. The work of McCauley et al. [29] was initiated from comprehensive investigation of burning characteristics of zirconium metal with air and barium chromate for the potential use of this reacting system in thermal batteries [16, 35]. The basic schematics of a thermal battery and key gasless and gas–solid SHS reactions are shown in Fig. 1.

Following this work on zirconium burning characteristics [35], McCauley and his co-workers [28–36] shifted their activities from using by-products of SHS to processing, focusing on the following: (i) utilize reaction sintering concepts without pressure, (ii) importance of physical and chemical characteristics of powders, (iii) focus on phase equilibrium, and (iv) detailed characterization of final sintered products.

Critical issues in reaction sintering are as follows:

- chemical driving forces much higher than conventional sintering,
- if gas forms most diffuse out,
- volume fractions of reactants and products change with time,
- Kirkendall effects: porosity formation due to density change between reactants and products,
- wetting between liquids and solid phases becomes important,

- grain size reduction from reactants—nucleate new phases.

A pioneering work of Holt and Kingman [36] was mainly focused on combustion synthesis of ceramic powders and refractory materials in general, which was more aligned with the research activities conducted in the former Soviet Union's laboratories.

A turning point in the United States efforts in SHS was catalyzed by a major contract from the Defense Advanced Research Projects Agency (DARPA) that was carried out during 1984–1986. The overall contract manager was J.W. McCauley and the program manager was J.B. Holt at the Lawrence Livermore National Laboratory with sub-contracts at The University of California, Davis, Ceramtec, Los Alamos National Laboratory, and Rice University.

The key universities, which started research in combustion synthesis in early 1980s included: University of California at Davis, Georgia Institute of Technology, State University of New York at Buffalo, and Northwestern University. These early research activities were supported by National Science Foundation, Department of Energy (Los Alamos National Laboratory and Sandia National Laboratory), and US Army. Also, some research in the area of combustion synthesis was conducted in US government laboratories, especially Department of Energy, US Army, and US Navy. In the table, the summary of research activities in academia, government laboratories, and industry in the United States at the end of 1980s is presented.

Both theoretical and experimental efforts were undertaken to explain various phenomena of combustion synthesis. Theoretical research describing combustion front stability and bifurcation analysis was done by Matkowsky from Northwestern University, and Margolis, Armstrong and Koszykowski from Lawrence Livermore National Laboratory. Professor Matkowsky has published numerous theoretical papers on the subject of gasless and gas–solid reactions [38–52]. His pioneering work with Margolis, Kaper, and Leaf on bifurcation on pulsating and spinning reactions in condensed two-phase combustion belongs to very fundamental classics of combustion synthesis [39]. His further analysis with Bayliss of two routes to chaos in condensed phase combustion as well as a series of theoretical papers on filtration combustion with Booty and scientists from Chernogolovka, Russia made very significant contribution to better understanding of complex nonlinear phenomena in combustion synthesis. Very accomplished mathematicians and theoreticians, such as Shkadinsky, Shkadinskaya, Aldushin, and Volpert from Russia cooperated closely with Professor Matkowsky during the 1990s. Dr. Volpert joined Northwestern University and he presently works there as a professor of applied mathematics. Professor Volpert published several papers with Professor Matkowsky on the theory of gasless and various aspects of filtration combustion in porous structures with and without deformation. He

also contributed to better understanding of combustion in microgravity environments and mathematical modeling of frontal polymerization and understanding of wave propagation during free-radical polymerization with the gel effect [53–63].

A parallel mathematical modeling effort was undertaken at the State University of New York at Buffalo under the leadership of Professor Hlavacek who joined that university in 1981. Professor Hlavacek established a very active research group which focused its research on both experimental studies and mathematical modeling of self-sustaining reactions and materials engineering aspects of combustion synthesis. Due to the access to parallel computer processors in mid 1985, his research modeling team was able to simulate complex combustion patterns, including transition to chaos, breaking of symmetry, fingering effects, multiple spinning waves in two and three dimensions, as well as complex behavior of the combustion front during gas–solid reactions [64–76]. Figure 2 shows the transition to chaos via period doubling in gasless reacting systems. A typical sequence of spinning combustion waves in two dimensions is shown in Fig. 3. It should be noted that these simulations were done using a very sophisticated adaptive mesh computer program, which allowed completing calculations on available supercomputers within a reasonable period of time. This computer technology looks old today, but truly it was the state-of-the-art twenty years ago.

In the 1990s, Professor Law from Princeton University published a number of papers describing model formulations, mathematical modeling of combustion front propagation and comparison of key combustion characteristics with experimental results [77–87]. Figure 4 shows comparison of experimental and theoretically predicted combustion limits for the Co–Ti system [82].

During the same period of time, other researchers from various universities also contributed to development of new reaction models and mathematical modeling of combustion synthesis processes. Contributions by Professors Munir, Stangle [88], Kanury [89], Bhattacharya [90], and Varma [91] are also of very significant importance.

The experimental research conducted in the US national laboratories and US universities resulted in many accomplishments, which led not only to significant contributions into the fields of physics, materials science, ceramic engineering, and reaction engineering but also to the development of several technologies, which resulted in their commercialization.

In academia, Professor Munir, one of the key SHS leaders in USA has been involved in the area of combustion synthesis from the early 80s. His research activities at University of California at Davis resulted in education of large number of excellent scientists who are

SHS R&D groups in the United States in late 80s

Organization	Principal investigators	Technology focus
Department of Defense		
Army Material Technology Lab.	Croft, Marzik, McCauley	Powder characterization, sintering, phase equilibria
Army Ballistic Research Lab.	Niiler, Kottke	Dynamic compaction, modeling
Army Research Office	Crowson	Coordination and management
Department of Energy		
Los Alamos National Lab.	Behrens	High-temp, chemistry, laser ignition, modeling
Lawrence Livermore National Lab.	Holt, Halverson, Chow	SHS, bulk materials, models
Sandia National Lab.	Margolis	Modeling
Academia		
Alfred University	Spriggs	Materials processing, reviews
Oregon State University	Kanury	Modeling
Washington State University	Wojcicki	Materials processing, eutectics
University of California Davis	Munir	SHS, materials processing, fundamentals
Northwestern University	Matkowsky	Mathematical analysis
Georgia Tech. Research Institute	Logan	SHS, materials processing, thermites
Rice University	Margrave	High temperature mass spectrometry
New Mexico Inst. of Mining & Tech.	Thadani	Explosive compaction
State University of NY Buffalo	Hlavacek, Puszynski	SHS, powders, mater, processing, math. modeling
University of California San Diego	Meyers	Explosive compaction
University of Florida	Clark, Dalton	Microwave processing
Colorado School of Mines	Moore	SHS, intermetallics
Industry		
Research Triangle Institute	Mullins	Fibers and metal matrix composites
CERAMETEC	Cutler	SHS, powders, thermites
General Sciences Inc.	Zavistanos	SHS densification
System Planning Corp.	Frankhouser	Reviews and analyses
Lockhead Corp.	Hardt	SHS, sintering; phase equilibria
Corning Glass Works	DeAngelis	Reactive hot pressing
W.R. Grace	Rice	Materials processing
Advanced Refractory Technologies	Blakely	SHS powders, whiskers
Innovative Materials, Inc.	Puszynski, Hlavacek	SHS; nitride, boride, and carbide ceramics; intermetallics
Benchmark Structural Ceramics	Hida	SHS powders and whiskers
Powder Technologies, Inc.	Logan	SHS powders and bulk materials
Synergetic Materials, Inc.	Halverson	Advanced materials
Kiser Research, Inc.	Kiser	Soviet SHS technologies

working in many countries. His selected major research contributions are listed below [92–105]:

- combustions synthesis of refractory carbides, borides, silicides, nitrides, and intermetallic compounds (1980s),
- analysis of the role of thermal migration in pore formation during SHS synthesis (1990),
- theoretical analysis of the stability of self-propagating combustion synthesis waves, concept of SHS diagrams (1990–1992),

- use of the Boddinton–Laye mathematical analysis for direct determination of kinetic parameters during SHS (1992),

- analysis of the origin of porosity in SHS products (1993),

- the role of electric fields in SHS reactions: modeling and experimental work (1995–1998),

- separation of the thermal (Joule heat) from the intrinsic (electron wind effect) contributions of the field (current), work on electromigration has demonstrated

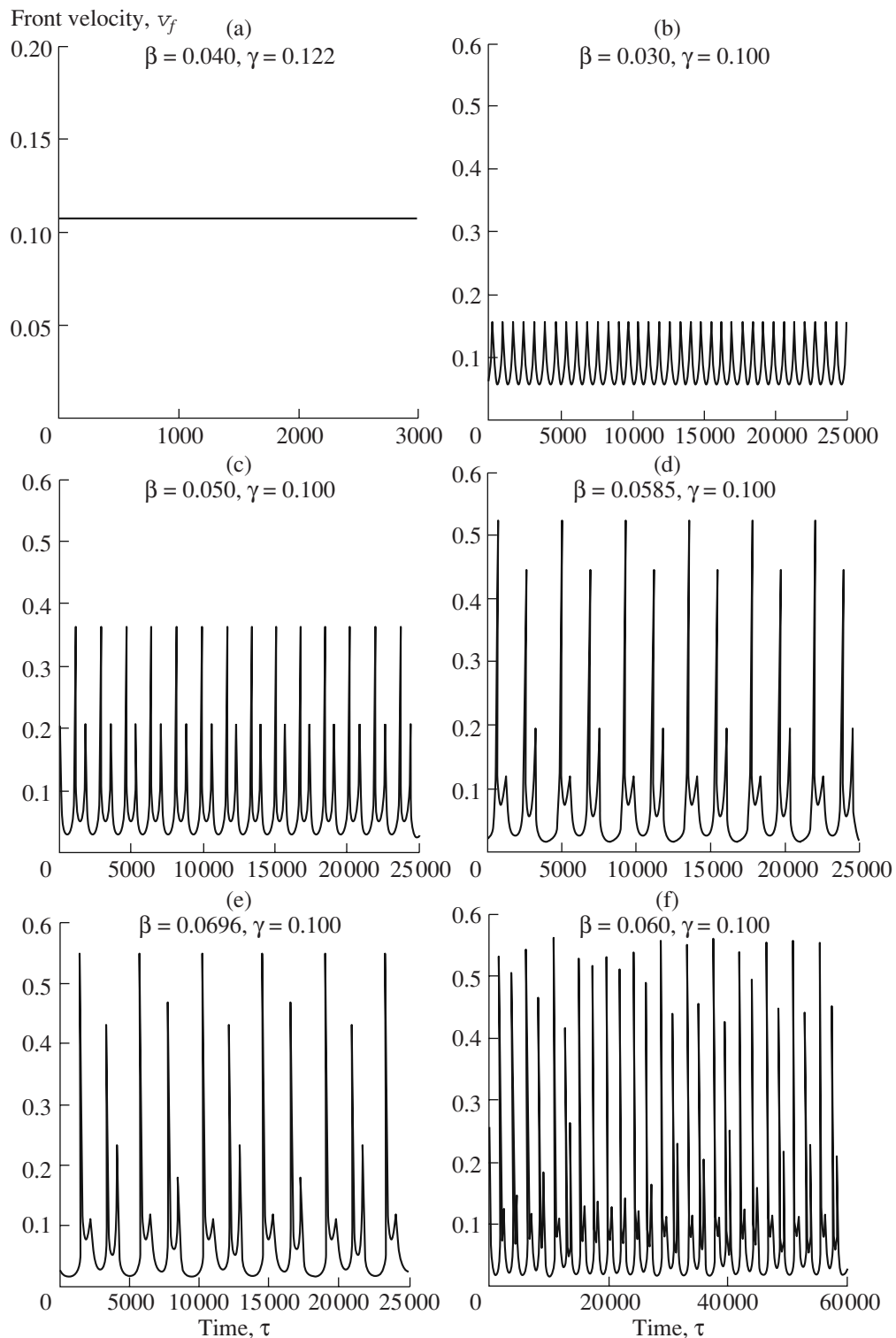


Fig. 2. Combustion front propagation velocity v_f in gasless systems with different dimensionless activation energy and heat of reaction vs. time τ [68].

field effect on point defect generation and mobility (2001),

- recent work on the combined mechanical and field activation to synthesize dense (bulk) nano-ceramics and nano-composites in one step (2001—present),

- use of field activation for simultaneous synthesis and consolidation of complex materials [Ti_3SiC_2 (1999), $\text{TiB}_2\text{-WB}_2\text{-CrB}_2$ (2001), AlN-SiC (1996–2000)],

- use of field activation for microalloying (2003–2004),

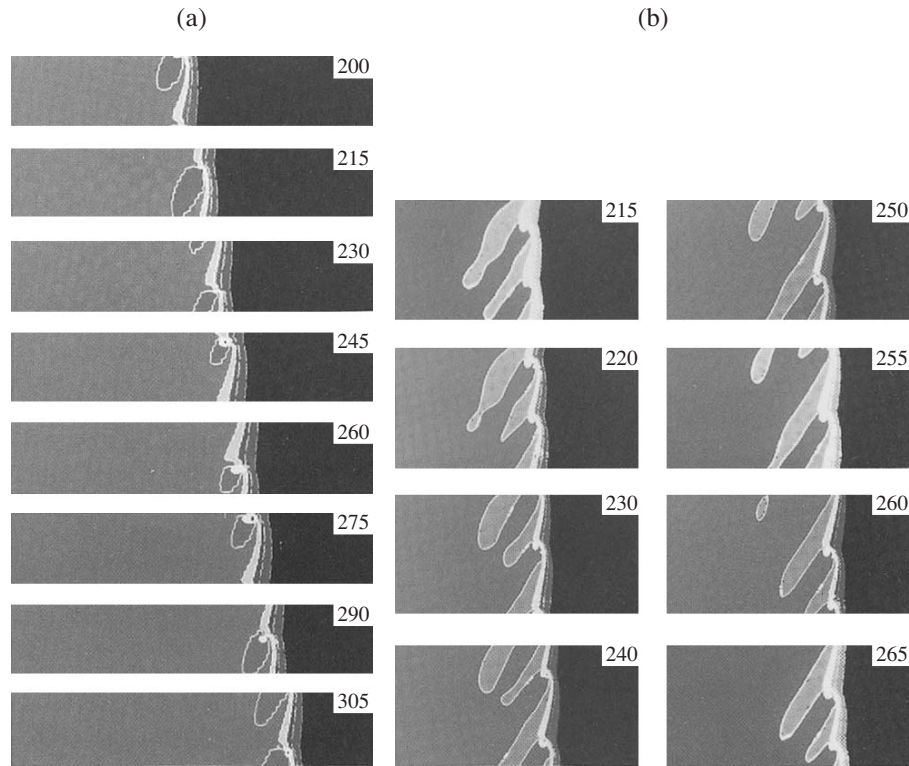


Fig. 3. Two-dimensional modeling: (a) single head spinning wave and (b) multiple head spinning waves [64].

- use of field activation to prepare nanostructured functional oxides for fuel cell applications: novel demonstration of power generation at room temperature by protonic conduction.

The main advantage of the field-assisted process is the electrical discharge at particle contacts which promote sintering. Numerous materials, including TiN, TiO₂, SiC, Si₃N₄-TiN, ZrO₂-Al₂O₃, and FeAl, were sintered during the past several years resulting in the formation of dense articles with nanosize grains. The starting powders were obtained by plasma, mechanical alloying, or sol-gel techniques. A very important modification of this field-assisted technique was presented by Munir [93]. It was demonstrated that the combination of field-assisted technique, such as SPS, and in-situ synthesis of materials from nanoreactants or mechanically activated powders may result in the formation of desired phase and consolidated products retaining nanostructure. Experimental results did show that the presence of electrical field influences on the mechanism and rate of the condensed phase reaction as well as the phase composition and elemental distribution in solid solutions. The main effects of the electric field during the reaction have been attributed to Joule heating, enhanced mass transport by electron-migration, and the formation of plasma on the particle level. Therefore, the entire process of in-situ densification of combustion synthesized bulk materials exhibiting a nanostructure can be divided into three steps:

- mechanical activation of participating reactants,
- cold compaction of pre-alloyed powders,
- field-activated pressure-assisted synthesis.

In the first step, reactant powders are mixed in a stoichiometric ratio and co-milled in a planetary mill in order to form nanocrystallites. During the milling, the particles are flattened, fractured, and welded. This process of grain size reduction, generation of residual stresses, and phase transformation has a significant effect on the kinetics of combustion reactions during the final consolidation step in the presence of electrical field.

The second step involves cold-compaction of mechanically activated powders into a graphite die. The final step includes simultaneous application of electric current and uniaxial pressure under the inert atmosphere. In this step, the combustion reaction is initiated by Joule heating and the hot product is densified within a few minutes. Relative densities between 90–100% of the theoretical density can be commonly achieved.

It should be mentioned that Professor Munir has published many papers and obtained numerous patents for his innovations of combustion synthesis. In this review only few selected papers are mentioned [92–105]. He has also made very important contribution to the SHS community by reviewing articles on SHS for many journals, including the *Ceramic Bulletin*, and *Materials Science Reports*, which are cited by thou-

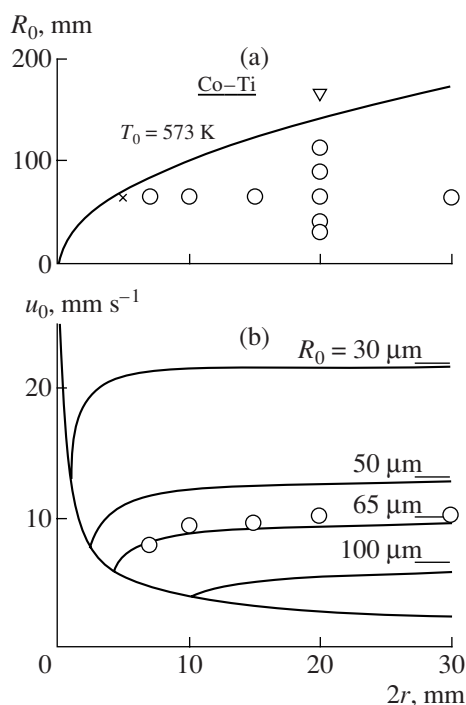


Fig. 4. Nanodiabatic combustion behavior for the Co-Ti system with stoichiometric mixture at $T_0 = 573$ K: (a) Range of flammability as a function of $2r$ and R_0 , data are from Itin et al.; (○) designates the steady propagation, (◻) the flame extinction during the propagation, and (×) the non-ignition. (b) Burning velocity u_0 as a function of $2r$, with R_0 taken as a parameter, data points are experimental in the literature.

sands and continue to be cited to the present. Professor Munir has established among US scientists the strongest collaboration with researchers around the world. He has collaborated with Professors: Frederic Bernard, University of Burgundy, Dijon, France; Manshi Ohyanagi, Ryukoku University, Seta, Japan; Umberto Anselmi-Tamburini, University of Pavia, Italy; Giacomo Cao, University of Cagliari, Italy; Manfred Martin, University of Aachen, Germany; Rainer Telle, University of Aachen, Germany; In-Jin Shon, Chonbuk National University, Korea; Myeong-Woo Cho, Inha University, Korea; Roberto Tomasi, Sao Carlos Federal University, Brasil; Qing-sen Meng, Taiyuan University of Technology, China; K.A. Khor, Nanyang Technological University, Singapore; Z.Y. Fu, Wuhan University of Technology, China; and Yu. Maksimov, Tomsk University, Russia. He has also ongoing collaboration with US national laboratories, including collaboration with Dr. Alex Gash from Lawrence Livermore National Laboratory, USA and Dr. John Neal from Oak Ridge National Laboratory, USA. Professor Munir has published many papers and he was awarded with numerous patents related to combustion synthesis. In 1993, he established the American Consortium of Combustion Synthesis.

The State University of New York at Buffalo (SUNY/Buffalo) was the second university strongly involved in combustion synthesis research. As indicated before, Professor Hlavacek built a very large group of PhD students and research scientists. His integrated approach resulted in a strong development of combustion synthesis technologies supported by strong basic experimental research and mathematical modeling programs [106–117]. In the mid 80s, Drs. Hlavacek and Puszyński successfully transferred the technology of synthesizing aluminum nitride by combustion synthesis technique into Advanced Refractory Technologies Company located in Buffalo, NY. This company was the first to produce aluminum nitride by this technique. In the late 1980s, other technologies for synthesis of silicon nitride, titanium carbonitride, α - and β -sialons, titanium carbide-titanium boride and silicon nitride-silicon carbide composites as well as tungsten carbide and aluminum phosphide were developed by Drs. Hlavacek and Puszyński. The university spin-off company Ceramic Materials Processing, Inc. was involved in manufacturing of ceramic and intermetallic powders by SHS method, scale-up of combustion reactors, and technology transfer. During the 80s and early 90s, several researchers visited SUNY/Buffalo. Dr. Puszyński joined Professor Hlavacek's group in 1982. In 1991, Puszyński accepted a position at the South Dakota School of Mines and Technology where he has been continuing SHS-related work. His research has been focused on combustion synthesis of nanopowders and nanocomposites as well as the reaction kinetics in systems consisting of nanosize reactants [118–126]. Professor Puszyński established close cooperation with Yerevan State University in Armenia, Academy of Mining and Metallurgy in Cracow, Poland and several US national laboratories. His recent work indicates that various intermetallic composites reinforced with single wall carbon nanotubes can be formed in a self-sustaining regime with the ultimate grain structure being at the nanoscale (see Fig. 5). His comprehensive work on combustion synthesis in the Si-Al-Ti-O-N-C system has led to the formation of many complex compounds with different morphologies and phase compositions. His work on chemically-assisted gas transport combustion synthesis led to successful synthesis of nanosize silicon carbide. Figure 6 shows inert gas pressure regimes where silicon carbide can be formed. Figure 7 shows different morphologies of silicon nitride formed with and without the presence of gas-transport promoting additives.

Professor Puszyński has been actively involved in the organization of technical sessions dedicated to combustion synthesis at various conferences, including the American Institute of Chemical Engineers and the American Ceramic Society. Professor Puszyński also serves as a frequent reviewer of journal manuscripts. He also serves as a consultant to Noveltec Co. in Tennessee, which is involved in production of variety prod-

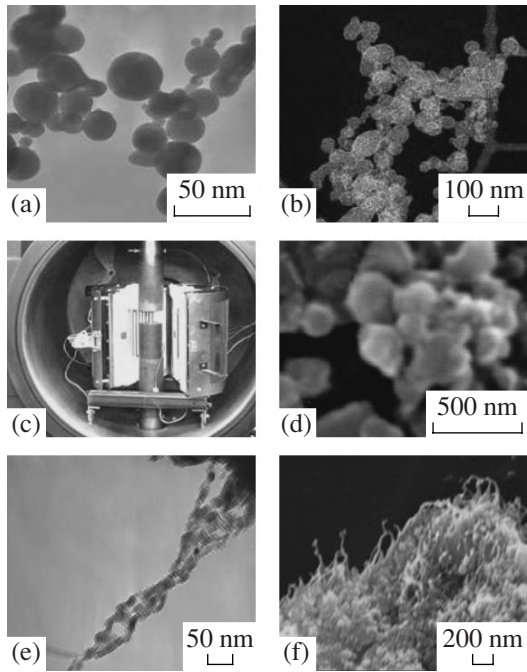


Fig. 5. (a), (b) TEM images of Al and Ni nanoreactants, (c) reaction chamber, (d) SEM image of nanosized nickel aluminate-alumina composite prepared by simultaneous combustion synthesis and densification, (e), (f) SEM images of single-walled carbon nanotubes reinforced nickel aluminate-alumina nanocomposites.

ucts, including sialons, carbides, borides, nitrides, and sulfides by the SHS technique.

Dr. Viljoen spent several years at SUNY/Buffalo in the late 1980s and early 1990s. Dr. Viljoen work in the SHS area was focused on fundamental aspects of combustion reactions involving the solid state. After accepting a professor position at University of Nebraska, he continued his fundamental work focusing on solid-solid reactions with mechanical coupling, understanding of solitons and non-equilibrium reactions in solid phases, combinatorial approach to surface contacts in solid-phase reactions, and analysis of the effect of heat transfer on combustion front propagation limits [127–131]. Professor Viljoen also contributed to a better understanding of strongly exothermic reaction taking place under strong compression. He also cooperated with Russian scientists, including Dr. Scheinberg, and he supervised several Russian graduate students who joined his research group.

Dr. Lis joined Professor Hlavacek's research group in the late 1980s. His research at SUNY/Buffalo was focused on combustion synthesis of silicon nitride-silicon carbide composites and sialons. He published jointly with Professor Hlavacek and his key staff several papers, which outlined key aspects of combustion synthesis, processing, and sintering of SHS synthesized materials. After his return to Poland, he continued

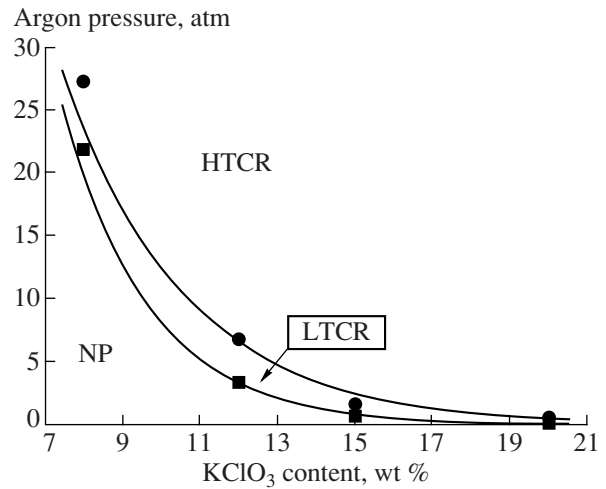


Fig. 6. Combustion propagation diagram in Si-C-KClO₃ reacting system: NP means no propagation; LTCR stands for low temperature combustion regime; and HTCR, for high temperature combustion regime.

building SHS related programs together with his former PhD advisor Professor Pampuch. Later, the group headed by Professors Pampuch and Lis became one of the most active European groups outside the former Soviet Union. It should be clearly noted that Professor Hlavacek educated many excellent Ph.D students who are currently working in the industry or academia. He also was the pioneer who introduced many chemical engineers into the field of combustion synthesis.

In the late 1980s and at the beginning of 1990s, several other US universities got involved in combustion synthesis research. In the early 1990s, Alfred University under the leadership of Drs. Spriggs and McCauley initiated a research program focusing on further development of SHS technologies. With the strategic hiring of Dr. Stangle, several R&D initiatives were conducted, including: (i) fabrication of dense MoSi₂ and MoSi₂-based composites using SHS process, (ii) combustion synthesis and fast-firing of nanocrystalline yttria-stabilized zirconia, (iii) fabrication of functionally gradient materials by SHS method (see Fig. 8 [156, 157]), (iv) development of a centrifugal SHS process and analysis of its fabrication capabilities, (v) investigation of the mechanism and kinetics of combustion synthesis, and (vi) study of the combustion synthesis process for materials fabrication. This multi-year research program resulted in thirty-one publications and international recognition of an established research center [132–162].

In addition, the Alfred group established several international collaborations:

- interactions and formal agreement with NRIM, Japan (Kaieda),

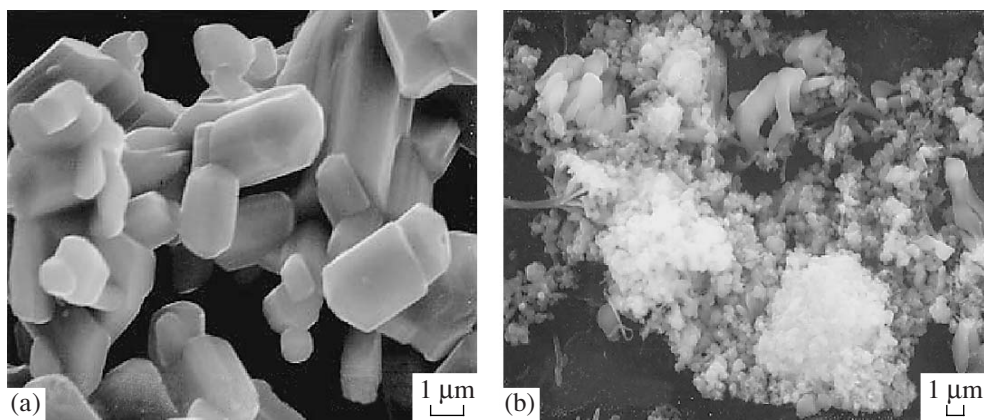


Fig. 7. SHS produced β - Si_3N_4 (a) and α - Si_3N_4 (b).

- formal agreement with the Institute of Materials Science, School of Mining and Metallurgy, Poland (Pampuch),

- interactions and formal agreement with ISMAN (Merzhanov and Borovinskaya).

At approximately the same time period, Professor Varma initiated combustion synthesis research at Notre Dame University. His initial research interest was focused on mathematical modeling of combustion fronts. However, very quickly his research evolved toward experimental investigation of reaction kinetics of heterogeneous reactions as well as understanding of system heterogeneity and melting effects on propagation of combustion fronts in the condensed phase [163–177]. Professor Varma invited a few Russian scientists, including Drs. Mukasyan and Rogachev, to work with

him at Notre Dame University. He also attracted several graduate students, including some from Russia. Dr. Mukasyan was offered a permanent position at this university and he is still working there conducting his own research program in the area of combustion synthesis. A few years ago, Professor Varma accepted a new challenging position at Purdue University where he continues research in the area of strongly exothermic non-catalytic reactions. Both Professors Varma and Mukasyan when working together at Notre Dame University conducted combustion synthesis research in a microgravity environment. They also investigated possibilities of synthesizing biomaterial using the SHS technique. They also initiated work on combustion solution of oxide nanomaterials for development of catalysts. Presently, Professor Mukasyan is actively continuing that research. A variation of the combustion synthesis process, namely utilization of exothermic redox reactions in solutions, was already investigated by several researchers in India and in the USA. Professor Bhaduri was among first who explored this technique in USA [178–182]. This type of the reaction is called solution combustion synthesis (SCS) and involves a self-sustaining reaction between metal nitrates and carbonaceous fuels, such as urea, glycine or carbohydrazide. The reaction between such fuel and oxygen containing species results in a significant heat generation. In practice, this process is accomplished by dissolution of metal nitrates and uniform mixing of the fuel and nitrates in water, preheating of the oxidizer-fuel solution with subsequent water vaporization, followed by self-ignition of the dry reactants. As a result, the formation of crystalline oxide nanopowders with tailored compositions can be formed. The main advantage of this approach is mixing of reactants at the molecular level. The overall reaction process is very fast and results in the formation of nanograins exhibiting a high purity due to vaporization of all volatile species at high reaction temperatures generated by this exothermic reaction. Another important advantage of this method is a possibility of the formation of complex oxide nanop-

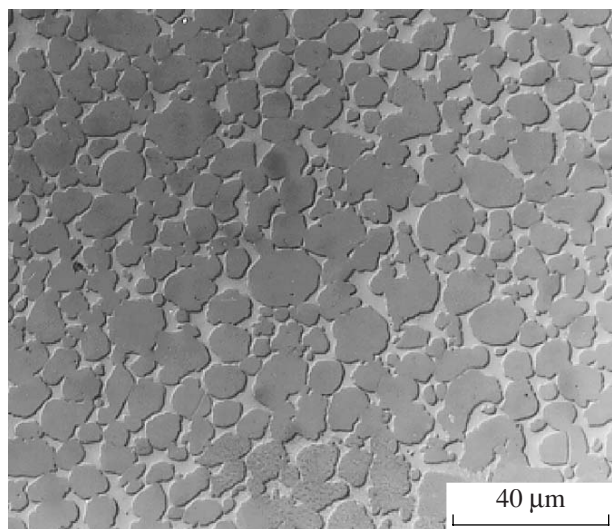


Fig. 8. Microstructure of in-situ densified TiC–25% Ni composite formed during combustion synthesis and obtained in Prof. Meyers' laboratory.

owers for different applications as structural ceramics, catalysts, bio- or fuel cell materials [183–188].

The combustion synthesis research at Colorado School of Mines has been carried out by Professor Moore for almost twenty years. Professor Moore's research interest has been on the formation of composite materials at normal or reduced gravity environments. The recent research interest of Professor Moore is focused on the formation of biomaterials [189–192]. Professor Moore is very actively involved in numerous professional societies and his published contributions into the field of SHS are highly regarded by the international SHS community.

A significant research effort in the USA was focused on simultaneous combustion synthesis and hot pressing. Professor Logan from Georgia Institute of Technology established an experimental program focusing on densification of titanium diboride and various composites generated during aluminum thermal reduction of oxides [193]. Professor Logan developed a strong cooperation with the R&D group led by Dr. Niiler from US Army Ballistic Laboratory and McCauley of the US Army Materials Technology Laboratory. Niiler and his co-workers were involved in shock densification of combustion synthesized materials by means of explosives [194, 195].

Shock-induced densification of ceramics and cermets by unique high speed forging was conducted by Professor Meyers and his research group at University of San Diego, CA [196–211]. Professor Meyers contributed to elucidation of the reaction mechanism at the front in the Ti–C system. This work was done with Dr. LaSalvia from the US Army Research Laboratory and produced some outstanding results describing physicochemical mechanism of that reaction [200, 201]. Professor Meyers also contributed to fundamental understanding of densification by quasi-isostatic pressing (QIP) of reaction products. This work was done in collaboration with Professor Olevsky [210, 211]. The use of a granular pressure transmitting medium, initially introduced at Chernogolovka, was used to produce TiC plates with dimensions of $12 \times 12 \times 2$ inches. Production and densification of TiC–NiTi cermets was another accomplishment of this technology. Figure 8 shows a typical microstructure of TiC–25%Ni composite material formed by SHS dynamically densified material. Professor Meyers collaborated with Dr. Kim, South Korea, Professor Meyer, Chemnitz University, Germany, Dr. Ramas Raman from Ceracon, Professor Olevsky, San Diego State University, and Dr. Jamet from Ecole Centrale de France.

A significant contribution into the area of shock densification of combustion synthesized intermetallics and ceramic materials was also made by Professor Thadani [212–217].

Recently, Professor Luss and his co-workers developed a novel efficient synthesis method named carbon combustion synthesis of oxides (CCSO) for production

of advanced nano and submicron complex oxides such as ferroelectrics (BaTiO_3 , SrTiO_3), hard and soft magnetic materials (Ba, Sr, Pb, Mn–Zn and Ni–Zn ferrites), superconductors (Y_{123}), optoelectronics (ZnSnO), solid-oxide fuel cell components (LaGaO_3), battery electrodes (LiMn_2O_4), catalysts, membranes, and digital pigments [218–221]. The method is a modification of self-propagating high temperature synthesis (SHS) that uses carbon as the heat generating fuel instead of a pure metal. The concentration of the carbon in the reactant mixture enables control of the moving front temperature and average temperature front velocity as well as the products particle size and surface area. CCSO may be used to produce oxides even when SHS cannot be applied, such as when the pure metal is pyrophoric (such as Li or La) or that it melts at room temperature (for example, Ga), or when the metal heat of combustion is relatively low. In contrast to the common SHS, the combustion product (carbon dioxide) is not incorporated into the product and exits from the sample. Moreover, the lubricating properties of carbon enhance the mixing by ball milling. The high rate of CO_2 release increases the porosity of the particles and the friability of the powder. The process is significantly faster than common calcinations processes and produces powders with smaller particle size.

Another interesting activity conducted by this group is focused on spontaneous magnetization generated by solid state combustion [222–228]. Using a highly sensitive high- T_c superconducting quantum interference device (SQUID), they were able to conduct the first measurement of the very low intensity (order of nT) transient magnetic field formed by a combustion front motion. The front propagation generated a slowly oscillating magnetic field on which, in some cases, high frequency small oscillations were superimposed. The magnetic power spectra of the oscillations scaled as a power law, suggesting that they are associated with a stochastic process. The combustion synthesis of ferrites generated qualitatively different magnetic fields under different modes of combustion front motion i.e., planar, spin, and pulsating. The average magnetization vector generated by either planar or pulsating combustion was oriented at a smaller angle with respect to the pellet axis ($\phi \leq 45^\circ$) than those generated by spin combustion ($60^\circ \leq \phi \leq 80^\circ$). The Earth's magnetic field had no impact on the spontaneous magnetization field of the samples. Dr. Luss' research group also developed a simple electromagnetic model which predicted the qualitative features observed in the experiments. The transient evolution of this field depends on whether the combustion temperature exceeds or does not exceed the Curie temperature. Figure 9 shows a case in which a residual magnetic field of about $4 \mu\text{T}$ was generated by the spontaneous magnetization of the ferromagnetic product $\text{PbFe}_{12}\text{O}_{19}$ in the post-combustion zone. The characteristic spontaneous magnetic field saturation time of about 250 s was much longer than the 1–2 s

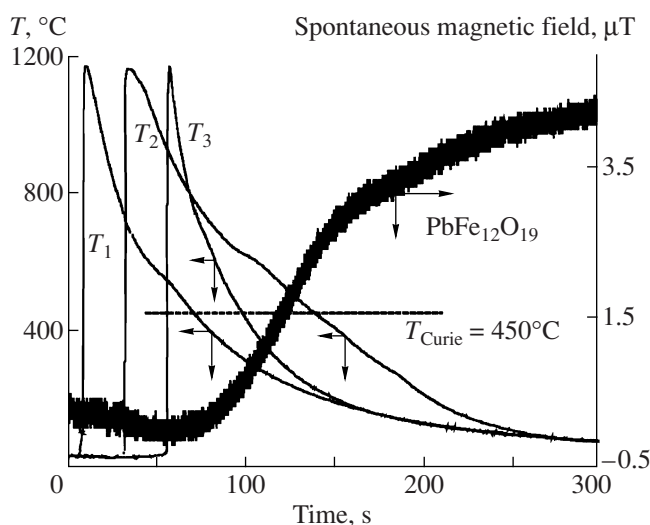


Fig. 9. Three temporal combustion temperatures in the top (T_1), middle (T_2), and bottom (T_3) of the sample and the spontaneous magnetic field measured near the one side of the sample during the combustion synthesis of ferrite. The distance between surface and sensor was 10 mm [226].

duration of the electrical signal. The magnetic field was created by three different mechanisms: (i) orientation of the magnetic dipole moments by internal electrical field force, (ii) dipole self-orientation along existing residual field of the bulk material during the cooling, and (iii) via chemisorption of O_2 molecules on the ferromagnetic surface.

During the past several years, another interesting technology was developed by Dr. Weihs from John Hopkins University [229–231]. Multilayer reactive foils provide ideal sample geometries for studying different SHS reaction with a high level of contact between reactants. In addition, the thickness of each layer can be precisely adjusted. Currently, his technology is used by Reactive Nanomaterials Co. for bonding dissimilar materials and in other applications.

SUMMARY

This review clearly indicated that the contribution of US scientists to both theoretical understanding of combustion reactions in condensed phase and development of new innovative technologies based on the principle of self-propagating reactions between solid reactants or those involving solid and gas interactions is significant. Despite the relatively small number of researchers involved in this field, the number of publications, patents, as well as technological know-how development is quite impressive.

Forty years have passed since the discovery of SHS in 1967. It should be emphasized that during the last seventeen years the exchange of information among all scientists working in combustion synthesis is without the political barriers that existed until the early 1990s.

Every two years, researchers have the opportunity to present their results at international SHS symposia. New close cooperation agreements have been established between different universities and research institutes. We hope that this trend will continue and new generations of scientists and engineers will contribute into the field of SHS freely and without any external constraints.

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