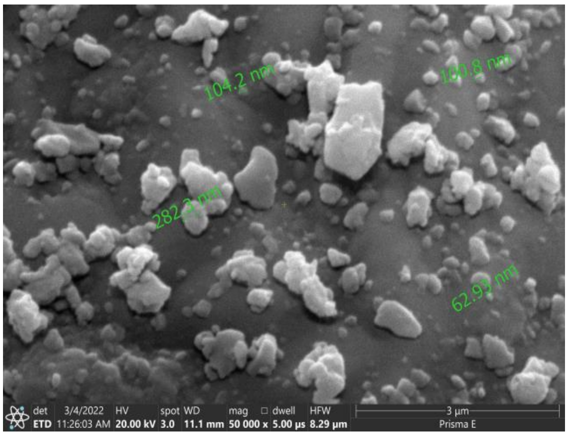
Laboratory of Technology of SHS processes

In 1975 in the IChPh of AS of Arm. SSR, in the laboratory of high-temperature synthesis***, the SHS processes in the Me-H_2 system were predicted and implemented for the first time.*** This direction became a priority for the studies of the Laboratory.

As a result of systematic study of the processes of combustion of metals in hydrogen and deuterium in the SHS mode, more than 200 binary and multicomponent hydrides and deuterides of transition metals were synthesized. The interaction of transition metals with hydrogen occurs with the release of heat, which ensures the propagation of the combustion front over an unheated tablet of the metal and its hydrogenation.

For example**:** **Ti + H_2 ↔ TiH_2 + Q (ΔH formation of TiH_2 - 39 kkal/mol)**

TiH_2



The microstructure of powders TiH_2;

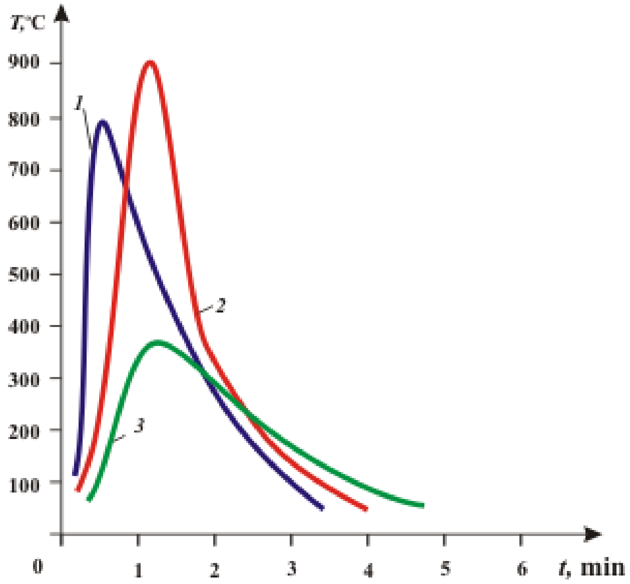
Various hydrogen-containing systems were investigated, including: Metals of III, IV and V groups, rear-earth metals-hydrogen, deuterium;

- Metals of IV and V groups - carbon hydrogen;
- Metals of IV and V groups - nitrogen - hydrogen;
- Zr_2Ni, Zr_2Co, ZrNi, ZrCo, Ti_2Co and other intermetallics-hydrogen.

As a result of systematic study, the scientific bases of SHS processes, proceeding in various systems in hydrogen atmosphere, were developed:

a) the factors controlling the character and propagation speed of the combustion wave, the temperature and completeness of combustion were revealed;

b) a two-stage mechanism of combustion and formation of hydrides in the SHS mode is demonstrated: in the first stage, a solid solution of hydrogen in the metal is formed in the combustion front, in the second stage, the hydrogen saturation occurs (after-hydrogenation), resulting in formation of hydrides of stoichiometric compositions.



Thermograms of processes of combustion of Ti (1), Zr (2) and ZrCo (3) intermetallic in hydrogen at 3 atm

c) **new interesting phenomena and effects, previously unknown in SHS processes, are discovered and investigated:**

- combustion in the low-temperature invisible spectral range of 250-350°C;
- critical phenomena at combustion of such complex systems, as Me-C-H, associated with the competition between reactions of hydrogenation and carbidization, which results in the change of the combustion mode;
- **For the first time,** the theory of Khaikin and Merzhanov is experimentally confirmed. This theory concerns the existence of non-uniqueness of stationary combustion modes in the systems with parallel reactions, in which the rate and temperature of combustion, at other equal conditions, depend on the conditions of ignition.
- **For the first time**, the SHS process of combustion of metals in the mixture of two reacting and competing gases, nitrogen and hydrogen was realized. It was shown that at combustion of Ti-N-H and Zr-N-H, depending on the ratio of partial pressures of gases, the combustion mode (the hydrogenating and nitriding reactions) interchanged; the mechanism of combustion and formation of high hydrogen content hexagonal (hcp) titanium and zirconium hydrido-nitrides was revealed. It was also found that the solid solutions of nitrogen in titanium and zirconium interact with hydrogen in the combustion mode without preliminary crushing and form the hydridonitrides.

d) **For the first time** it was shown that in hydrogen not only high-dispersed metal powders, but also the metal sponge and such waste as chips can burn. This is important for development of hydride manufacturing processes. These studies allowed to develop a technological process for utilization of titanium, zirconium, hafnium and other metal waste using the HC and SHS methods.

e) The physical-chemical characteristics (chemical and phase composition, crystal structure, thermal stability, decomposition kinetics, etc.) of the synthesized hydrides were studied.

f) For the first time it was shown that during synthesis by burning in hydrogen, self-purification of the final product occurred.

The results of study of hydrogen containing multicomponent systems indicate the complex nature of the synthesis reactions. They involve several competing processes that can be regulated. Using the optimal modes of their realization, it is possible to synthesize the materials of given chemical and phase composition. Important is that this method allows synthesis in one technological stage from the elements directly. The large possibilities of SHS method in the synthesis of complex hydrogen-containing compounds were demonstrated. The SHS method turned to be a very effective for the synthesis of binary hydrides and deuterides of transition metals of III, IV, V groups and lanthanides (tables 1), hydrides of intermetallics (**Zr_2CoH_5; $ZrCoH_3$; $ZrNiH_3$** etc.), carbohydrides and nitridohydrides of titanium and zirconium (**$TiC_{0.4}H_{1.2}$; $ZrN_{0.3}H_{1.5}$** etc.), hydrogen containing complex refractory materials. (Fig.1). More than 200 hydrides and **deuterides** were synthesized .

Table 1. Characteristics of several binary hydrides and deuterides

Metal	Content of $H_2(D_2)$, wt. %	Crystal lattice	Lattice parameters, Å	Calculated formula
Sc	4.25 3 .01	FCC FCC	a=4.782 A=4.698	ScH_2 $ScD_{0.73}$
Y	3.255 4.41	HCP FCC	a=3.661; c= 6.630 a=5.197	$YH_{2.9}$ $YD_{2.1}$
Ti	4.01 7.03	FCC FCC	a=4.460 a=4.51	TiH_2 $TiD_{1.82}$
Zr	2.16 4.16	Tetragonal	a=3.527; c= 4.476 a=3.520; c= 4.476	ZrH_2 $ZrD_{1.96}$
Hf	1.09 2.11	Tetragonal	a=4.911; c= 4.405 a=4.911; c= 4.405	HfH_2 $HfD_{1.93}$
V	1.71	Tetragonal	a=3.310; c= 3.339	$VH_{0.8}$
Nb	0.95	Orthorhombic	a=4.451; b=4.878; c=3.453	NbH
Nd	1.78 3.61	FCC FCC	a=5.446 a=5.364	$NdH_{2.6}$ $NdD_{2.5}$
Sm	1.87	HCP	a=3.771; c=6.782	SmH_3
Ho	1.78	HCP	a=3.653	HoH_3
Gd	1.79	HCP	a=3.373; c=6.71	$GdH_{2.88}$

Table 2. Characteristics of intermetallics and their hydrides

Compound	Content of H_2,wt. %	Crystal lattice, Parameters, Å	Temperature range of dissociation, °C
Zr_2 Co Zr_2CoH_5	- 2.02	Tetragonal, a=6.387; c=5.542 Tetragonal, a = 6.906; c=5.550	- 190 –360
ZrCo $ZrCoH_3$	- 1.68	Cubic, a = 3.197 Orthorhombic, a=3.37; b=10.57; c=4.318	- 200 – 370
Zr_2 Ni Zr_2NiH_5	- 2.08	Tetragonal, a=6.54; c=5.340 Tetragonal, a=6.86; c=5.657	- 170 – 250
Zr Ni $ZrNiH_3$	- 1.96	Orthorhombic, a = 3.29; b = 9.998; c = 4.080 Orthorhombic, a = 3.53; b = 10.62; c = 4.328	- 170 – 250
Ti_2 Co Ti_2CoH_3	- 1.7	Cubic, a=11.31 Cubic, a=11.89	- 240 – 360

Table 3. Characteristics of carbohydrides and nitridohydrides.

Formula	Content, wt. %			Crystal lattice, Parameters, Å	Temperature range of dissociation, °C
	H_2	C	N_2		
$TiC_{0.4}H_{1.2}$	2.2	8.45	-	hcp, a = 3.09; c =5.089	400 –840
$TiC_{0.45}H_{0.5}$	0.95	10.08	-	fcc, a = 4.296	380 – 840
$TiC_{0.5}H_{0.5}$	0.9	10.39	-	fcc, a = 4.296	425 – 800
$TiC_{0.6}H_{0.4}$	0.68	12.72	-	fcc, a = 4.30	760 – 810
$ZrN_{0.3}H_{1.5}$	1.55	-	3.81	hcp, a = 3.27; c =5.519	370-795
$TiN_{0.28}H_{1.33}$	2.2	-	7.6	hcp, a = 3.044; c =5.09	455- 610

Hydrides of transition metals have unique physical-chemical characteristics. Hydrogen, embedded into crystal lattice of a metal, can radically change the properties of the latter. Hydrides of transition metals and alloys are of great value as condensed hydrogen carriers. Interest in hydrogen and metal hydrides is associated with such global problems as the environmental and the depletion of fossil fuels. The spectrum of application of hydrides is very diverse: in hydrogen energy they serve as components of environmentally pure engine fuel (hydrogen storage); in the nuclear power - as materials protecting against radiation and as reactor materials, in the chemical industry - as catalysts; in powder metallurgy - for producing finely disperse powders of transition metals; etc.

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12. S.K. Dolukhanyan, N.A. Martirosyan, S.S. Simonyan, B.A. Galchenko, I.P. Borovinskaya, A.G. Merzhanov. The way of obtaining powders of carbides of titanium and zirconium. Author's certificate No. 1387313. Bull. № 13, 1988
13. A.G. Aleksanyan, S.K. Dolukhanyan, A.G. Hakobyan, S.K. Dolukhanyan, G.K. Gevorkyan, M.A. Kuzenkova, A.G. Merzhanov. Method for obtaining molybdenum aluminosilicide. Author's svid. No. 1429525. Bull. № 37, 1988.
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16. S.K. Dolukhanyan, A.G. Akopyan, R.A. Karimyan, N.L. Mnatsakanyan. Method for the preparation of transition metal hydrides. Author's certificate № 1513813. Bull. № 47, 1989
17. P.S. Kisly, V.V. Ivzhenko, M.A. Kuzenkova, Ya.A. Kryl, A.G. Merzhanov, I.P. Borovinskaya, V.K. Sulzhenko, S.K. Dolukhanyan, A.G. Aleksanyan, L.S. Popov. Charge for high-quality ceramic material. Author's Certificate No. 1520826. Bull. No. 41, 1989.
18. N.N. Aghajanyan, S.K. Dolukhanyan. Method for obtaining niobium carbide. Authors' Certificate No. 1522655. Bull. № 42, 1989.
19. P.S. Kisly, V.Yu. Kodash, M.A. Kuzenkova, A.G. Hakobyan, S.K. Dolukhanyan, A.G. Merzhanov, F.S. Garibyan, G.K. Gevorgyan, S.S. Torosyan, M.B. Gutman and I.V. Voronkin. Material for the electric heater. Author's certificate No. 1718698, Bull. № 9, 1992
20. S.K. Dolukhanyan, A.G. Akopyan, A.G. Aleksanyan, N.N. Aghajanyan, R.A. Karimyan, N.L. Mnatsakanyan Method for obtaining nitrogen-containing alloys. Author's certificate № 1827394, Bull. № 25, 1993.

Participation in international symposiums and conferences

1. All-Union Conference "Combustion processes in chemical technology and metallurgy", (USSR) 1973, Chernogolovka.
2. All-Union II Conference on Technological Combustion. (USSR) Chernogolovka 1978.
3. V-th International Symposium on Combustion Processes. Krakov (Poland), 1977.
4. V1-th International Symposium on Combustion Processes. Karpacz (Poland), 1979,
5. All-Union IV Symposium on Macroscopic Kinetics and Chemical Gas Dynamics, (USSR) Chernogolovka, 1978.
6. All-Union Conference on Rare and Sparse Elements. Armenian SSR, Tsaghkadzor, 1981.
7. All-Union Conference "High-temperature physics-chemical. processes at the solid-gas interface ", Zvenigorod, M, Nauka,1984.
8. I All-Union Symposium on Macroscopic Kinetics and Chemical Gas Dynamics, Chernogolovka, 1984.
9. I-st Int. Symposium Self Propagating High Temperature Synthesis, 23-28 Sept., 1991, Alma-Ata.
10. 9-th World Hydrogen Energy Conference. 22-25 June 1992. Paris-France, 1992.
11. International scientific and technical seminar "Metal-hydrogen-92", September 15-19. Donetsk, 1992.
12. III Inter-republican conference "Hydrogen Materials Science and Chemistry of Metal Hydrides, ICHMS-93, Katsiveli, Crimea, Ukraine October 8-15,1993.
13. NATOInt.Conference "Hydrogen Material Science and Chemistry of Metal Hydrides", 2-8 Sept. 1995, Katsiveli, Crimea, Ukraine,
14. 11-th World Hydrogen Energy Conference. Stutgart, Germany 23-28 June 1996.
15. Int.Symp. on Metal-Hydrogen Systems. Fundamentals and Applicatopn. Les Diablerets, Switzerland, 1996.
16. MH1998, Int.Symp. on Metal-Hydrogen Systems. Fundamentals and Applications. 4-9 Oktober 1998, Hangzhou, CHINA
17. VI Int. Conference «Hydrogen Materials Science and Chemistry of Metal Hydrides», Katsiveli, Yalta, Ukraine, Sept. 2-8, 1999
18. V Int. Symposium on Self-Propagating High-temperature Synthesis (SHS-99), Moscow, Aug.16-19, 1999
19. Int. Symposium on Metal Hydrogen Systems, MH2000, 1-6 Oct. 2000, Noosa, Queensland, Australia,
20. Int. Seminar "Conversion Potential of Armenia and ISTC Programs, Yerevan, 2-7 October 2000
21. ICHMS`2001, VII International Conference "Hydrogen Materials Science and Chemistry of Metal Hydrides". 16-22 September 2001, Alushta, Crimea, Ukraine

22. Int. Symposium on Self-Propagating High-Temperature Synthesis (SHS-2001) 14-18 October, 2001 Haifa, Israel
23. Int. Symp. on Metal Hydrogen Systems. Fundamental and Applications. Annecy, France, Sept.2-6, 2002.
24. International Symposium BB "Defect Properties and Related Phenomena in Intermetallic Alloys", USA, Boston MA, MRS - 2, December 2-6, 2002
25. World European Powder Metallurgy Association, the Exhibition “EuroPM2005”,12-th International Exhibition and Conference of Powder Metallurgy, 2-5 Oct., 2005. Prague, Czech Republic
26. Int.Conference ”Nonisothermal Phenomena and Processes”,Yerevan, Armenia, Nov. 27 – Dec.1 2006,
27. Euro PM2006 Congress and Exhibition, Ghent, Belgium, 23-25 Oct. 2006, European Powder Metallurgy Association
28. X Int..Conf. “Hydrogen Material Science and Chemistry of Carbon Nanomaterials” ICHMS`2007, Sudak, Crimea , Ukraine, 22-28 Sept. 2007
29. Int. Symposium on Metal-Hydrogen Systems MH2008, Reykjavik, Iceland, June 24-28, 2008
30. X Int.l Symposium on Self- propagating High-temperature Synthesis, SHS-2009, Tsakhkadzor, 6-11 July, 2009, Armenia
31. ICHMS`2009, “Hydrogen Material Science and Chemistry of Carbon Nanomaterials”,
32. XI Int..Conf., Yalta-Crimea-Ukraine, August 25-31, 2009
33. Int. Symposium "Metal-Hydrogen Systems. Fundamentals and Applications", MH2010, Moscow, Russia, July 19-23, 2010
34. 17th Int. Conference on Solid Compounds of Transition Elements, SCTE 2010, September 2-10, 2010, Annecy, France
35. The 7th International School-Conference of Young Scientists and Specialists, October 24-28. 2011, Zvenigorod, "Interaction of hydrogen isotopes with constructional materials"
36. XI Int. Symposium of Self-Propagating High Temperature Synthesis, 5 - 9 Sept. 2011, EDEN Beach Resort Hotel, Anavyssos, Attica, Greece
37. The 7th International School-Conference of Young Scientists and Specialists, October 24-28, 2011, Zvenigorod.
38. "School-conference" Interaction of hydrogen isotopes with constructional materials ", Moscow 2011.
39. Int. Symposium Nuclear Magnetic Resonance in Condensed Matter, NMRCM2011, 27 June – 1 July, 2011, St-Petersburg, Russia, (2011).
40. Int. conference "Modern problems of chemical physics", posv. 50th anniversary of the IHF them. A.B. Nalbandyan of the NAS of RA, October 9-12. 2012.
41. III Int. Conference on Chemistry and Chemical Technology, Yerevan, 16-20 Sept. 2013
42. IV conference of the Armenian Chemical Society (adjacent to participation) "Achievements and problems", October 7-11. 2014 Yerevan-Vanadzor.
43. International symposium "Rare and rare-earth elements: extraction, separation and modern materials" August 21-26, 2014 Yerevan, Armenia.
44. IV International. conference "Contemporary problems of chemical physics", 5-9 October 2015, Yerevan.

3. Scientific projects and grants

Date	Project Title	Principal Investigators
2022	**21AG-2F059**. Development of a technological process for the synthesis of multicomponent, multifunctional alloys of refractory metals and intermetallic compounds, promising as modern construction materials and hydrogen accumulators	S.K.Dolukhanyan G. N. Muradyan
2022	**1-22/TB-22. Applied (commercial) grade titanium based alloys synthesis by "Hydride Cycle" method**	D. Mayilyan
2020	**20DP-1D03**. Development of SHS technologies for titanium, zirconium hydrides and their intermetallic compounds as high energy additives or components of reactive materials	N.N. Aghajanyan
2016	**ISTC A-2287**, Formation of structure of hydrogen induced aluminides of IV-V group metals in hydride cycle and study of their physical - chemical properties , Status 3 “Approved without funding”	S.K.Dolukhanyan O.P.Ter-Galstyan
2014	Contract ChPh-30062014, CONTRACT ON RESEARCH AND DEVELOPMENT, Zecotek Dispaly Systems Pte Ltd, a company incorporated in Singapore. Development of a new generation of hydrogen-containing materials, including nanomodified ones,	S.K.Dolukhanyan, A.G. Aleksanyan

	based on refractory metals, alloys and intermetallic compounds of groups III-IV-VIII, for additive 3d technologies	
2009	**ISTC A-1794**, The using of SHS and "hydride cycle" techniques for synthesis the alloys of transition metals with high hydrogen adsorbing properties, as effective hydrogen storage. Status 3 - **"Approved without funding"**	S.K.Dolukhanyan, A.G. Aleksanyan
2009-2013	**IAEA Research Contract No15720/R0:** The receiving of alloys of transition metals with high hydrogen adsorbing properties using SHS and "hydride cycle" techniques and application of nuclear methods for their microstructural characterrization.	S.K.Dolukhanyan, A.G. Aleksanyan
2006-2008	**ISTC A-1249 -** Synthesis of Metal Hydrides, "Self Propagating High Temperature Synthesis of Hydrogen Content Materials with Maximum High Content of Hydrogen"-	S.K.Dolukhanyan, A.G. Aleksanyan
2003	**ISTC #A-575** "Synthesis and investigation of hydrogen containing materials used in biological protection from ionizing radiation" , Status 3 "Approved without funding"	S.K.Dolukhanyan
1998-2001	**ISTC A-192 -** Electron Beams for Hydride Formation, "The Influence of Electrons Beams on Formation of Binary and Multicomponent Hydrides with Extremal Properties"	S.K.Dolukhanyan