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SYSTEM PLANNING CORPORATION

**A WORKING PAPER:
SOVIET DEVELOPMENTS
IN REGARD TO
SUPERPLASTICITY IN
CERAMIC MATERIALS**

SPC 849

September 1982

William L. Frankhouser

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I. EXECUTIVE SUMMARY

A. INVESTIGATIVE BACKGROUND, OBJECTIVE, AND SCOPE

A review of recent literature on the superplasticity phenomenon in ceramic materials and an assessment of relevant Soviet technology development have been performed by System Planning Corporation (SPC) for the Materials Sciences Division (MSD) of the Defense Advanced Research Projects Agency as Task No. 15 under Contract MDA903-82-C-0025. The objective in performing the review was to determine if Soviet work in this field represented a major national development program.

Superplasticity is a physical state whereby structural materials can be plastically formed or shaped beyond conventionally accepted limits for the specific conditions employed. Researchers have also shown that drastic changes in other physical properties (e.g., electrical and magnetic) may accompany the manifestation of superplasticity. In the case of ceramic materials, the superplastic state might be used to produce more intricate structural shapes and/or to simplify high-temperature production operations. In comparison to most metallic structural materials, ceramics are much more intractable to being formed, particularly when products with sharply changing contours are required.

This investigation was initiated when Soviet technical literature recently published as a posthumous honor to G. V. Samsonov [Ref. 1] indicated that the superplasticity phenomenon in ceramic materials might be receiving considerable attention in the U.S.S.R. Since Samsonov had been a foremost Soviet materials authority, particularly in development of refractory products, these references to superplasticity could have implied that a significant Soviet national development program had been organized to explore this phenomenon. Major technology breakthroughs in production of

ceramic materials could lead to important defense applications, and a decision was made to investigate further. Thus, the objective has been to determine whether these references to superplasticity were simply offhand remarks or if they pointed to a major materials research and development (R&D) effort.

The initial investigative plan was to review references to superplasticity in international computerized technical data bases and thereby to identify significant R&D programs in that field. Unfortunately, more than 1,000 references are available in the best-known technical data bases, and the overwhelming majority refers to the superplasticity phenomenon in metallic materials--which has been under rigorous study in several countries over at least four decades. As a result of this large number of references, the main investigation was restricted to a search for Soviet references to superplasticity in ceramic R&D. The search was reorganized by concentrating on key Soviet researchers in the field and checking their publications in three computerized data bases. The technology assessment is based on surveillance of more than 100 abstracts of international technical literature and on an assessment of approximately 20 Soviet technical articles that have some bearing on plasticity properties in ceramic materials.

B. SUMMARY OBSERVATIONS

1. A comprehensive Soviet R&D effort is under way to improve the ductility property in complex ceramic compounds (ternary and binary combination compositions).

Soviet researchers have been examining a large number of complex binary-combination and ternary ceramic compounds in a search for mechanisms to improve ductility in refractory materials at ambient temperatures. Enhanced ductility is valuable in production of improved hard alloys (for use in machine tools) and abrasives. Multiphased ceramic compounds that provide a desirable balance between hardness and ductility properties represent the Soviet answer to these needs for better industrial products.

Some of these same complex compounds exhibit the superplasticity property in the temperature range where they are hot formed into product shapes or sintered for densification, but development objectives do not emphasize superplasticity per se.

2. Industrial-defense applications of the technology developed with complex compounds could result in improved ceramic products and lower processing costs.

Soviet research appears to be on the verge of industrial applications for complex ceramic compounds in production of machine tools and abrasive products. The major motivations envisioned for those applications are improved performances, reductions in cost of machining, grinding, and polishing operations and replacement of strategic tungsten metal. Another adaptation of the technology may be to use the superplasticity property in forming intricate lightweight refractory ceramic products that were not previously possible. Soviet researchers have obtained up to 30 percent plastic deformation in binary combination compositions where only 5 to 6 percent had been achieved with the pure single-component compositions. This increased plasticity may be effectively utilized in hot forming and sintering operations.

3. The status of domestic R&D in this area of ceramic technology should be investigated.

The existence of similar U.S. development activities has not been explored in this investigation. A cursory review of Dialog data base printouts suggests that major U.S. ceramics research programs probably do not include specific tasks to exploit industrial applications of ceramic compounds with complex compositions. A detailed examination of the literature and a survey of industrial research programs is needed to confirm or disprove this impression.

This assessment of the Soviet R&D program should be sent to selected U.S. organizations that are engaged in ceramic R&D and/or production. Return comments should be requested concerning possible existence and development status of any domestic efforts that may parallel this work in the U.S.S.R.

II. TECHNOLOGY REVIEW AND ASSESSMENT

A. IDENTIFICATION AND REVIEW OF RELEVANT SOVIET RESEARCH

The technical articles on Soviet research in ceramic materials relevant to superplasticity were identified by reviewing three computerized data bases and recent issues of two Soviet technical journals. The data bases, published by Dialog Information Services, Inc., are:

- Dialog File No. 8, Compendex--engineering information from 1970 to present.
- Dialog File No. 32, Metadex--science and practice in metals and materials research from 1966 to present.
- Dialog File No. 94, Scisearch--literature of science and technology from 1978 through 1980.

The Soviet technical journals that contain most of the pertinent information are:

- Neorganicheskie Materialy, Izvestiya Akademii Nauk SSSR
- Poroshkovaya Metallurgiya.

The subject of superplasticity in ceramic materials has been mentioned in descriptions of R&D performed at the following three research locations in the U.S.S.R.:

- Institute of Materials Science in the Ukrainian, S.S.R, including the N. K. Krupskaya Kherson Pedagogical Institute
- M. V. Lomonosov Moscow Institute of Fine-Chemical Technology
- Lensovet Leningrad Technological Institute.

In some instances, researchers at the Moscow institute listed above have cooperated in this field with the Chirchik Branch of the All-Union Scientific Research Institute of Hard Alloys and have probably cooperated in

relevant research with the All-Union Scientific Research Institute of Abrasives and Grinding. The major thrusts of relevant research tasks at these three locations vary, but they seem to be related by a common thread. The common technological thread is that all of the tasks involve complex compositions of ceramic compounds, rather than simple compounds, and that all are oriented to improving either ductility or formability and densification of ceramics. Improved ductility is considered to be a significant factor in producing materials used as tool bits and abrasives, and increased high temperature creep (plasticity) may be significant in simplifying the hot pressing and sintering operations often used in production of refractory ceramic materials. The research subjects, lead researchers, and ceramic materials involved are listed in Table 1 for several Soviet R&D groups that appear to have been most active in this field in recent years.

B. ASSESSMENT OF SOVIET RESEARCH ACTIVITIES

Although G. V. Samsonov of the Institute of Materials Science had considerable influence in setting the Soviet course in ceramic research over recent years, relevant literature does not indicate that one of his basic research objectives was to achieve superplasticity in ceramic materials. The research emphasis appears to be on achieving more ductility and plasticity in ceramic materials. Essentially all of the researchers have related improved ductility and plasticity to complex compound compositions (e.g., ternarys such as $Ti(C,N)$, $(Ti,Cr)B_2$, and Ti_2AlC or binary combinations such as $TiC + ZrC$), $(ZrB_2 + ZrN)$, and $(Y_2O_3 + AlN)$. Although the superplasticity phenomenon has sometimes been manifested in such complex compounds, it was not the original motivation for the research. In 1973, G. V. Samsonov emphasized the need to understand "...the nature of the strength and plasticity of refractory compounds...in order to produce better constructional and abrasive materials..." [Ref. 2]. In that article he also mentioned "...the recently discovered phenomenon of superplasticity of complex carbides..." [Ref. 3], indicating that superplasticity was not an experimental objective in the research of the early 1970s.

TABLE 1.
SOVIET RESEARCH GROUPS INTERESTED IN DUCTILITY OR SUPERPLASTICITY
IN CERAMIC MATERIALS

<u>ORGANIZATION</u>	<u>PROBABLE RESEARCH LEAD PERSONS</u>	<u>RESEARCH SUBJECTS</u>	<u>RESEARCH COMPOUNDS</u>
Institute of Materials Science	T. Ya. Kosolapova	Ductile cutting tools and abrasives	Ti ₂ AlN, Ti ₃ AlN, Ti ₂ AlC, Ti ₃ AlC
		Improved abrasives, ternary compounds; ternaries exhibiting superconductivity and wear resistance	Ti(C,B) Ti ₂ AlN
	M. S. Koval'chenko	Hot-pressed binary carbides are wear resistant	(Ti,Cr)B ₂
	G. A. Bovkun	A binary wear-resistant diboride	(Nb,Zr)B ₂
M. V. Lomonosov Institute	R. A. Andrievskii, I. I. Spivak	Superplasticity and high temperature creep are ex- amined to determine under- lying causes and to develop new hard alloys (cemented carbides)	M ₁ ¹ N ₁ ¹ + M ₂ ² N ₁ ¹ , M ₁ ¹ N ₁ ¹ + M ₁ ¹ N ₂ ² , M ₁ ¹ N ₁ ¹ + N ₁ ¹ , M ₁ ¹ N ₁ ¹ + M ₂ ² N ₂ ² , M ₁ ¹ N ₁ ¹ N ₂ ² , (see foot- note a)
Lensovet Institute	S. S. Ordan'yan	Inter-relationships of superplasticity, compres- sive creep, and sintering	M ₁ ¹ N ₁ ¹ N ₂ ² M ₁ ¹ N ₁ ¹ +M ₂ ² N ₂ ² , M ₁ ¹ N ₁ ¹ +M ₂ ² N ₁ ¹

^aM represents various metallic elements, and N represents non-metallic elements.

During the 1970s and into the early 1980s, the research motivation in this field at the Institute of Materials Science was the development of improved cutting tools and abrasives. During 1976, emphasis was placed on ternary compounds of Ti-C-N, Ti-Al-C, and Ti-Al-N because of the combination of high hardness and good ductility [Refs. 4, 5, and 6]. Titanium carbonitrides also showed considerable promise as alternatives to standard cemented carbides (hard alloys), and titanium aluminonitrides and carbides were being investigated for use as abrasives. These latter materials were proven to retain their abrasive power for longer periods in polishing glass than diamond, aluminum oxide, and conventional simple carbide abrasives. During 1978 and 1980, reports were published on densification and high temperature creep of titanium-chromium diborides (ternary compound compositions), that "...surpass other borides in hardness and in wear and oxidation resistance..." [Refs. 7 and 8]. During 1979, another article [Ref. 9] indicated that ternary titanium carborides (with TiC and TiB₂ phases) had better abrasive capacity than unalloyed titanium carbide. In 1980, other researchers at the Institute of Materials Science cited the "...urgent need to seek new binders for alloys based on transition metal borides..." and reported success in hot pressing (Nb,Zr)B₂ alloys that would serve in wear-resistant applications [Ref. 10]. Sintered hard alloys of these materials were reported to have "...high hardness, good ductility, and therefore considerable potential as wear-resistant materials...." The wear resistance of Nb-40 mol percent ZrB₂ was claimed to be comparable to that of standard WC-TiC-Co alloys.

During the same period, researchers at the Institute of Fine Chemical Technology were examining superplasticity as made evident in high temperature creep of complex ceramic compounds. Evidence of superplasticity was reported in a number of carbide compounds and in several other cases, as shown in Table 2. In 1974, these researchers reported that the common feature in superplasticity mechanisms in carbide alloys was the diffusional nature of the phenomenon [Ref. 11]. In another instance, when working with TiB₂-TiC and ZrB₂-ZrN, the researchers reported that extremely fine phases and approximately equal concentrations of phases contributed to superplasticity [Ref. 12]. Possible superplasticity was also reported in the

TABLE 2.
CERAMIC COMPOUNDS REPORTED BY SOVIET RESEARCHERS
TO EXHIBIT THE SUPERPLASTICITY PHENOMENON

<u>Carbides</u>	<u>Other Combinations With Carbides</u>	<u>Others</u>
NbC-C	Ti(C,N)	AlN-Y ₂ O ₃
TiC-C	TiC-TiB ₂	ZrB ₂ -ZrN
ZrC-C	TiC-ZrB ₂	
TiC-VC		
TiC-ZrC		
VC-CrC		
VC-HfC		
VC-ZrC		

AlN-Y₂O₃ system in 1976 [Ref. 13]. Studies of underlying causes for superplasticity included grain boundary contaminants, interstitial alloying, grain structures, and electronic structures [Refs. 14 and 15]. One researcher concluded that "...practically any two-phase fine-grained composition can be superplastic if it contains isodiametral inclusions with maximum interphase boundaries...." The most important factor in achieving superplasticity was considered to be prolonged preservation of fine grains, but electronic structure was also considered to be of some significance in assessing the role of interphase energy. In 1980, this investigative knowledge was being used to develop titanium carbonitrides as alternatives for tungsten carbide in production of cemented carbides (hard alloys) [Ref. 16]. During this same period, the physiochemical properties of titanium carbonitrides were also being explored at the All-Union Scientific-Research Institute of Abrasives and Grinding [Ref. 17]. This research location possibly indicates that near-term production applications were envisioned.

During the 1970s, researchers were examining the compressive-creep property in complex ceramic compounds at the Lensovet Technological Institute, and the superplasticity phenomenon was reported and quantitatively characterized. The discovery of superplasticity in ceramics has been attributed by Soviet authors to these researchers [Ref. 3], and early in the decade they were measuring high-temperature creep in the ZrC-NbC system

[Ref. 18]. By mid-decade they were also working with the binary TiC_x-TiB_2 system [Ref. 19] and examining brittleness in stoichiometric and non-stoichiometric compositions of niobium carbide [Ref. 20]. During 1977, a published article indicated that possible superplasticity had been encountered in the $HfC-HfB_2$ system at the eutectic composition [Ref. 23]. During 1979, articles were published on sintering densification of non-stoichiometric titanium carbide [Ref. 22] and on the creep property of zirconium carbide that contained varying levels of tungsten carbide [Ref. 23]. During 1980, a report was published that emphasized attainment of the superplastic state (in the form of high rates of compressive creep) in the $ZrN-ZrB_2$ and $TiC-TiB_2$ systems [Ref. 24]. These researchers suggested that superplasticity may be "...characteristic of fine-grained two-phased materials..." and that good deformability and thermal fatigue resistance were also exhibited in the ceramic systems that were investigated. In one series of tests on $ZrC-ZrB_2$, strains of 30 percent were obtained without rupture at temperatures of 2,300°C. This contrasts with maximum strains of 5 to 6 percent attainable in the pure components (ZrC and ZrB_2) under the same conditions. The researchers attribute much of this large gain in strain with the complex compounds to phase boundary slip. Maximum gain in strain was attained when the phase-volume ratios were approximately 1 : 1 and particle sizes of each phase were not more than 2 microns.

C. SUPERPLASTICITY RESEARCH OVERVIEW

The foregoing assessment suggests strongly that the superplasticity state is not an objective of Soviet research in ceramic compounds. The Soviets are, in fact, searching diligently for mechanisms by which ceramic materials can be made more ductile over wide temperature ranges. Industrial utility of ceramics in cutting tools and in abrasive applications is considerably enhanced when an optimized combination of hardness (strength) and ductility is achieved. The Soviet researchers have also learned that thermal superplasticity often accompanies attainment of the desired balance in the strength-ductility relationship. The major benefit of the superplasticity phenomenon is envisioned in simplification of sintering and hot-

forming operations, and Soviet research is under way in both of these technology areas.

Soviet research into underlying causes of both enhanced ductility and superplasticity is continuing. Generally, this research and the theories under investigation are similar to those being studied in regard to superplasticity in metals. In a recent Soviet publication on superplasticity in metals [Ref. 25], the deformation mechanisms were identified as:

- Grain boundary slide
- Directional diffusion mass transfer
- Intergranular slide.

The current state of Soviet thinking in regard to superplasticity mechanisms in ceramics is similar as noted in the foregoing assessment of research programs.

The potential industrial-defense significance of this Soviet research in complex ceramic compounds can be grouped into two categories--potential cost reductions and new lightweight products. If the researchers are successful in developing better hard alloys (for use in machine tools) and abrasives, costs of machining, grinding, and polishing operations will be reduced. Furthermore, tungsten--which Soviet researchers class as a strategic material--can be conserved. In the lightweight product category, simplified high-temperature forming may yield products that have not been available in ceramic materials heretofore and which could be lighter in mass than their metallic counterparts. If the superplasticity property can be effectively utilized, shapes with complex contours might be formed directly where only machined components had been available previously. Certainly, any laboratory successes are likely to be manifested in hardware applications in the machine tool and abrasive products industries during the late 1980s.

The relative status of U.S. R&D in this field vis-a-vis the Soviet programs is difficult to assess. Although the literature review was not planned to examine U.S. progress, a cursory look at the Dialog printouts suggests that no major U.S. technology program is directed to exploring the industrial potential of complex compositions in ceramic compounds. In the

case of superplasticity, large numbers of references have been identified for both U.S. and U.S.S.R. metals research, but the number of Soviet articles is much greater in the case of ceramics research. In fact, only three U.S. papers on superplasticity research with specific ceramic materials have been noted [Refs. 26, 27, and 28]. An in-depth investigation of U.S. industrial ceramics research would be required to make a meaningful comparative judgment of relative progress in this field in the two countries.

One way in which this judgment could be made would be through an inquiry from MSD at the Defense Advanced Research Projects Agency to U.S. organizations engaged in ceramics R&D Activities and/or in manufacture of ceramic products. This inquiry could be accompanied by the foregoing assessment of the relevant Soviet R&D programs.

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