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Chapter 1

Introduction

Interest in superplasticity is extremely high. The major areas include superplasticity in metals, ceramics, intermetallics, and composites. Superplasticity at very high strain rates (i.e., approximately $0.1-1 \text{ s}^{-1}$) is an area of strong emphasis that is expected to lead to increased applications of superplastic-forming technology.

Historically, there has been no universally accepted definition for superplasticity. After some debate, the following version was proposed and accepted at the 1991 International Conference on Superplasticity in Advanced Materials (ICSAM-91) held in Osaka, Japan [1]:

Superplasticity is the ability of a polycrystalline material to exhibit, in a generally isotropic manner, very high tensile elongations prior to failure.

It is anticipated that there will continue to be some modifications to this definition, but it should serve as a working definition for a phenomenon that was scientifically reported in 1912 [2] and, indeed, may have a far longer history, as described in the following chapter.

During the course of the ICSAM–91 Conference [1], many different superplastic materials were described. A list of those mentioned is presented in Table 1.1 [3]. It is reasonable to infer from the broad range of superplastic materials listed that there is now a good basic understanding of the requirements for developing superplastic structures. This extensive list also indicates that research on superplastic ceramics and intermetallics has increased dramatically in recent years since the first superplastic ceramic was not observed until 1985 and the first superplastic intermetallic until 1987. The first paper [4] to appear on superplastic ceramics was presented at the 1985 conference in Grenoble, France (though no

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Table 1.1 Superplastic materials described during ICSAM-91 [1]

				Ceramics and ceramic
Metallic alloys and composites			Intermetallics	composites
Al-Ca-Si	IN 905XL	Tool steel	Ni ₃ Al	YTZP
Al-Ca-Zn	IN 9051, 52	UHC steel	Ni ₃ Si	YTZP/Al ₂ O ₃
AlCu	IN 100	Superdux 64	Ti ₃ Al	Hydroxyapatite
				$(Ca_{10}(PO_4)_6(OH)_2)$
Al-Cu-Mn	IN 625 LCF	Fe-Cr-Ni	TiAl	Si ₃ N ₄ /SiC
Al-Cu-Si	MA6000	SKD11.PM steel	α–2	Al ₂ O ₃
Al-Cu-Zr	MA754	Stainless steels	Super α -2	$3Al_2O_3 \cdot 2SiO_2$
Supral 100	Cu-Al-Ni	T15 PM HSS	Fe ₃ (Si,Al)	α' sialon
				$(Si_{\delta-\chi}Al_{\chi}O_{\gamma}N_{\delta-\chi})$
Supral 200	Cu-42Zn	HPb59–1 brass	Nb ₃ Al	β' sialon
				$M_{Z/N}Si_{6-X-Z}$
				$Al_{X+2}O_XN_{8-X}$
Al-Li	Coronze 328	Pb-62Sn	Ni ₃ (Si,Ti)	Si-Al-M-N-O
Al 8090	Cu–P	Zn-22Al	Ni-9Si	Al ₂ O ₃ :Pt (95:5)
Al 2090	Cu-Zn-Ni	Zn–Cu–Ti	Ti-34Al-2Mo	BaTiO ₃
Weldalite	Nb-Hf-Ti	α/β brass	Ni-Si-Ti(B)	ZnS
Al-Mg-Mr	n Ti–Mo–Sn–Zr	SiCp/7475 Al		ZnS/diamond
Al-Mg-Cr	Ti–9V–Mo–Al	$\alpha \operatorname{SiC}_{w}/2024\mathrm{Al}$		PbTiO ₃
Al-Mg-Zr	Ti-6Al-4V	$\alpha \operatorname{SiC}_{w}/2124\mathrm{Al}$		Fe ₃ C/Fe
A1 5083	Ti SP700	$\alpha \operatorname{SiC}_w/6061 \operatorname{Al}$		WC/Co
Al–Zn–Mg	Ti–36Al	SiC _w /7075Al		$YBa_2Cu_3O_{7-X}$
Al 7475	Ti–Al–Mo	$\alpha \operatorname{Si}_{3}N_{4(w)}/2124Al$		$YBa_2Cu_3O_{7-Y}X+Ag$
Al 7064	Ti IMI843	$\alpha \operatorname{Si}_{3}N_{4(w)}/7064A1$		
IN 9021	Mg-Mn-Ce	β Si ₃ N _{4(w)} /2024Al		
IN 90211	Mg–Li	β Si ₃ N _{4(w)} /6061A1		
IN 905XL	Mg–Al–Zr	SiC _p /6061Al		
		SiC _w /Zn–22Al		

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evidence for large tensile elongations was given in that particular paper); however, an elongation to failure of about 100% in polycrystalline MgO was reported in 1965 by Day and Stokes [5]. The 1991 Osaka conference was the first in the ICSAM series with papers on superplastic intermetallics.

An important conclusion that can be drawn from Table 1.1 is that historically simple concepts regarding key characteristics of superplastic materials are no longer appropriate [3]. For example, it was formerly believed that a superplastic material was a 'metallic two-phase material with a uniform, fine, equiaxed, grain (phase) size.' Clearly, the materials now shown to exhibit superplasticity are much broader than would be covered by this definition, and a more appropriate, although more complex, description is: 'Metallic, ceramic, intermetallic, or composite multiphase materials with uniform or nonuniform, relatively coarse (20 μ m) to ultrafine (30 nm) grain sizes that have isotropic or anisotropic grain (phase) shape, size, or orientation.'

Because the development of superplasticity is concerned with achieving high tensile ductility, the maximum elongations that may be attained in different types of materials is of great interest. The maximum elongation achievements to date are summarized in Table 1.2 [3], and this list covers not only the maximum elongations in different classes of materials, but other achievements related to material characteristics, fabrication of parts by superplastic forming, and activities within the scientific community. This list may be viewed as a guide against which to gage future trends in superplasticity research and technology.

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Table 1.2 Achievements in superplasticity

Property	Level of achievement	Material or contributor
Maximum superplastic elongation in a metal	8000%	Commercial bronze [6]
Maximum superplastic elongation in an intermetallic	>800%	Ti_3Al (super α -2)
Maximum superplastic elongation in a metallic composite	1400%	SiC _w /Al 6061 (thermal cycling) [7]
Maximum superplastic elongation in a ceramic	1038%	YTZP [8]
Maximum superplastic elongation in a ceramic composite	625%	YTZP/Al ₂ O ₃ [9]
Maximum superplastic elongation at a very high strain rate	1250% at 50 s ⁻¹	Mechanically alloyed aluminum IN 9021 [10]
Highest strain rate sensitivity	<i>m</i> =2	lpha' sialon, $meta''$ sialon, two-phase sialons [11]
Finest grain size in a consolidated material	30-40 nm	Al_2O_3/SiO_2 (9:1) and ZrO_2 (Y_2O_3 -stabilized)
Structural components manufactured per year	265 000	Superform, Ltd.
Longest continuous research career on superplasticity	over 33 years	Professor Presnyakov, Russia [12]
Largest (and only) institute for superplasticity	400-person level	Ufa, Russia

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Chapter 2

Key historical contributions

2.1 Before 1962

It is often thought that superplasticity is a relatively recent discovery. It is intriguing to speculate, however, that the phenomenon may have had its first application in ancient times. Geckinli [1], for example, has raised the possibility that ancient arsenic bronzes containing up to 10 wt% arsenic, which were used in Turkey in the early Bronze Age, could have been superplastic. This is because the materials are two-phase alloys that may have developed the required stable, finegrained structure during hand-forging of intricate shapes. Furthermore, the ancient steels of Damascus, in use from 300 B.C. to the late nineteenth century, are similar in composition to modern ultrahigh-carbon steels that have recently been developed, in large part, for their superplastic characteristics [2, 3]. A perspective of the historical context of superplastic studies with respect to the modern time frame is shown in Figure 2.1.

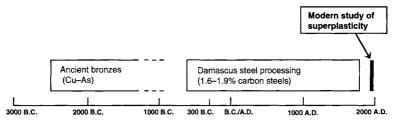


Figure 2.1 Historical perspective of the development of superplasticity from its possible ancient origins to the present time.

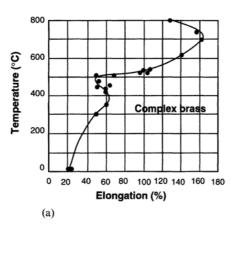
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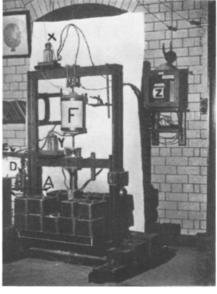
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A paper published by Bengough in 1912 [4] is believed to contain the first recorded description of superplasticity in a metallic material. Bengough describes how 'a certain special brass ... pulled out to a fine point, just like glass would do, having an enormous elongation.' The quote (which even today is a useful definition containing the essential feature of superplasticity) was made by Bengough in a written discussion following a paper by Rosenhain and Ewen [5] on the amorphous cement theory. Examination of Bengough's original work shows that the special brass was an $\alpha + \beta$ brass and exhibited a maximum elongation of 163% at 700 °C; the original data from Bengough's paper are shown in Figure 2.2(a). Considering the crude equipment (Figure 2.2(b)), poor temperature control, large gage length, and relatively high strain rate that was probably used in Bengough's experiment, his results are really rather remarkable. It is interesting to note that materials similar to those studied by Bengough have been processed to be superplastic in recent times. For example, Cu-40% Zn brasses have been developed for superplasticity at temperatures ranging from 600 to 800 °C [6].

Other observations of viscous-like behavior in fine-grained metals can occasionally be found in early literature. Jenkins, for example, achieved elongations of 300 to 400% in Cd–Zn and Pb–Sn eutectics after thermomechanical processing and in 1928 [7] provided the first photographic evidence of superplastic





(b)

Figure 2.2 (a) Data from the original paper by Bengough [4] in 1912 in which superplasticity was first documented scientifically. The elongation to failure of an $\alpha + \beta$ brass is plotted as a function of temperature and a maximum elongation of 163% is noted. (b) The equipment used by Bengough.

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Before 1962

behavior in a tested sample as shown in Figure 2.3. Interestingly, Jenkins did not find the observation to be important enough to include in either the synopsis or the conclusions of his paper. In 1934, Pearson [8] dramatically demonstrated, using a Bi–Sn sample that had been deformed to nearly 2000% and then coiled (as shown in Figure 2.4), that unusually large elongations could be achieved in certain fine-structured, two-phase materials. As a result, he is often credited with having first demonstrated superplasticity.

At a later date in East Germany, Sauerwald [9] reported that a number of aluminum- and zinc-based alloys exhibited good tensile ductility, and he patented several compositions for their formability. At the same time, or perhaps slightly earlier, work in the Soviet Union was underway to specifically address the phenomenon, and Bochvar and Sviderskaya [10] coined the term *sverhplastichnost* (ultrahigh plasticity) in their 1945 paper on superplastic alloys. Apparently the term *superplasticity* was used for the first time in the English language in Chemical Abstracts in 1947, but first appears in a 1959 technical paper by Lozinsky and Simeonova [11] on the subject of *Superhigh Plasticity of Commercial Iron under Cyclic Fluctuations of Temperature*. Some of the key observations and discoveries in superplasticity through this time period are summarized in Figure 2.5.

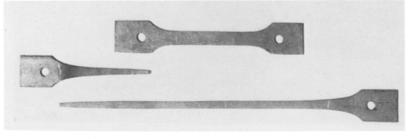


Figure 2.3 The first published photograph of a superplastically deformed sample by Jenkins [7] in 1928. The material is either a cadmium–zinc alloy or a lead–tin alloy (Jenkins does not specify which it is) and has undergone about 300% elongation.

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Key historical contributions

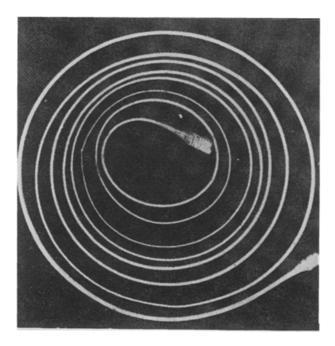


Figure 2.4 Pearson's famous photograph [8] in 1934 of a Bi–Sn alloy that has undergone 1950% elongation.

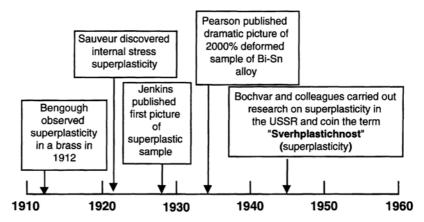


Figure 2.5 Key discoveries in superplasticity in the early and middle part of the twentieth century prior to major development of interest in the West during the 1960s.

From 1982 to the present

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2.2 From 1962 to 1982

Although papers on superplasticity occasionally appeared after 1945, the major increase in interest came in 1962 with a review article by Underwood [12] on work in the Soviet Union. A graph (Figure 2.6) was included in this review that illustrated the ductility of Zn–Al alloys after quenching from 375 °C. A maximum ductility of 650% was achieved at 250 °C for a 20% Al and 80% Zn alloy (the monotectoid composition). This remarkable result, achieved on a sample that required only a quenching treatment, attracted the attention of Backofen at the Massachusetts Institute of Technology. His research group [13] studied details of the Zn–Al monotectoid alloy and a Pb–Sn eutectic composition material.

Of great significance is that Backofen and his colleagues showed that the superplastic Zn–Al alloy could be formed into a practical shape by a simple airpressure operation, as in glassblowing [14]. An example of such an article is shown in Figure 2.7. This practical example, showing the spectacular formability of a superplastic metallic alloy, was probably the single biggest initiator of the rapid growth in the field of superplasticity that took place after the Backofen paper was published in 1964.

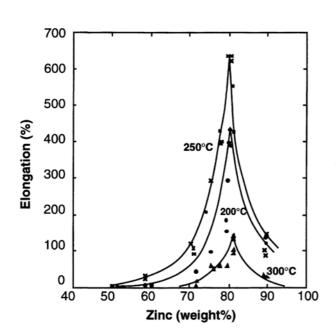
By 1968, just four years after the Backofen demonstration of superplastic forming, the first review paper on the subject was published by Chaudhari [15], and in the next year, the first book entiled *Superplasticity of Metals and Alloys* was published by Presnyakov [16]. After that, monographs by Western [2, 6, 17, 18], Soviet [19–21], and Japanese [22] researchers were published, as were numerous review articles [12, 23–25].

2.3 From 1982 to the present

A 1982 international conference entitled *Superplastic Forming of Structural Alloys* [26] showed how great the interest was in the subject of fine-structure superplasticity from both academic and commercial viewpoints. It was also the first conference fully dedicated to the subject of superplasticity. At this conference, the feasibility of the commercial application of superplasticity was reviewed for alloys based on titanium [27], nickel [28], aluminum [29, 30], and iron [31].

Since the 1982 conference, other significant international symposia have been held. Superplastic forming was the topic of a 1984 symposium held in Los Angeles [32]. A conference on superplastic aerospace aluminum alloys was held in Cranfield, United Kingdom, in July 1985 [33]. The second international conference on superplasticity was held in Grenoble, France, in September 1985 [34]. NATO/AGARD selected superplasticity as one of their lecture series in 1987 [35] and again in 1989 [36]; bilateral symposia on superplasticity between 10

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Key historical contributions

Figure 2.6 The ductility of Zn–Al alloys taken from the 1962 Underwood review article [12].

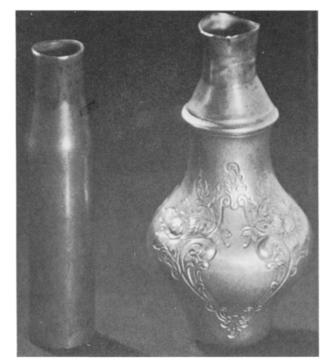


Figure 2.7 A salt-andpepper shaker superplastically formed by a simple gas-pressure operation in the Zn–Al alloy from Backofen *et al.* [13] in 1964.