Mines Branch Information Circular IC 235

SUPERPLASTICITY

by

H. M. Weld*

ABSTRACT

The superplasticity phenomenon, which is characterized by a metal's capacity for a large degree of relative uniform extension, is reviewed for the purpose of assessing both its potential applicability to practical metallurgical operations and the possible benefits to be derived from further research and development. Detailed are the requirements for a metal to be superplastic, as well as the conditions necessary for the phenomenon to occur. Twenty-nine metal systems reported to be superplastic are listed, including pure metals and single-phase and multi-phase alloys. A number of the methods are reviewed that are employed to generate the required fine grain structure--in the order of a micron--mechanically by extrusion or rolling, or thermally by eutectoid decomposition or compact sintering. It is concluded that superplasticity may be found in many other metal systems that possess a stable micrograin size within the limited temperature and strain-rate range in which a high strain-rate sensitivity can exist. Potential applications of superplastic metals by extruding, rolling, deep forming, coining, bulge-forming, and die-less wire drawing are reported. Possible problems in connection with the use of superplastic metals--such as oxidation, formation of voids, and strain-rate control--are discussed. Many of the commercial alloys reported to be superplastic will owe their acceptance to their improved mechanical and chemical properties rather than to their remarkable high-temperature ductility. The future application of the superplastic effect is viewed as being most successful when used to form large parts that are beyond the capabilities of most presses.

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Direction des mines, Circulaire d'information IC 235

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RÉSUMÉ

L'auteur étudie le phénomène de la superplasticité, qui se caractérise par l'aptitude d'un métal à s'allonger fortement d'une manière assez uniforme, afin d'évaluer à la fois son application possible aux travaux de métallurgie courante et les avantages éventuels à retirer de recherches ultérieures. Il décrit les paramètres de superplasticité d'un métal, ainsi que les conditions requises pour que le phénomène se produise. Il énumère vingt-neuf systèmes de métaux qui sont considérés comme superplastiques, y compris des métaux pûrs et des alliages à une ou plusieurs phases. Il passe en revue un certain nombre de méthodes employées pour produire la texture voulue, à grain fin (de l'ordre d'un micron), soit mécaniquement par extrusion ou par laminage, soit thermiquement par décomposition eutectoïde ou par agglomération des compactes. Il conclut que le phénomène de superplasticité peut se produire dans de nombreux autres systèmes de métaux dont la taille des micrograins est stable dans la limite de température et de vitesse de déformation où il peut exister une grande sensibilité à la vitesse de déformation. L'auteur énumère les applications des métaux superplastiques à l'extrusion, au laminage, à l'emboutissage profond, à la frappe de la monnaie, au bombage et à l'étirage des fils sans filière. Il examine divers problèmes que peut poser l'utilisation des métaux superplastiques, notamment l'oxydation, la formation de cavités et le contrôle de la vitesse de déformation. Un bon nombre des alliages commerciaux considérés comme superplastiques seront acceptés en raison de leurs propriétés mécaniques et chimiques améliorées plutôt que de leur remarquable ductilité à haute température. L'auteur juge que l'application éventuelle de la superplasticité atteindra sa plus grande efficacité dans la formation de grosses pièces qui dépassent les capacités de la plupart des presses.

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INTRODUCTION

A superplastic metal is characterized by its ability to stretch when hot to many times its own length, without necking, when slowly strained at low stress. Such behaviour is not commonly observed in metals, but is often observed in non-metals such as glass or plastic. The deformation of hot glass provides a typical example of the effect when a thick rod of hot glass can be gently pulled into a long thin thread many times its original length, without breaking. Most hot deformable metals have only a relatively short range of uniform plastic deformation before they begin to neck and, after further deformation, fracture.

Superplasticity was first reported in metals by Pearson(1) in 1934, when he stretched, in a hot extension test, an extruded wire of Bi-Sn eutectic alloy nearly 2000% before fracture. The term "superplasticity" was coined some ten years later by direct translation of a Russian term introduced by Bochvar and Sviderskaia(2) to describe similar mechanical behaviour of Al-Zn alloy of approximately eutectoid composition.

The phenomenon attracted the interest of other Russian scientists during the fifties, particularly Presnyakov(3,4) who with his co-workers studied alloys of Al-Zn, Al-Cu, Al-Si, Al-Ni, Al-Fe, Cu-Zn and Cu-Ni and reported superplasticity in eutectic or eutectoid systems but not in systems with continuous solubility. Earlier work by Russians and others, related to superplasticity, was reviewed in detail by Underwood(5) in 1962 and later by Backofen et al.(6).

These papers have stimulated a wider interest in the phenomenon and, since 1964, there has been increasing scientific involvement which is reflected in a rapidly growing number of scientific papers and technical sessions on the subject. Superplasticity was the topic for two sessions of the American Society for Metals at the 1967 Materials Engineering Congress, and three sessions of the Metallurgical Society of the AIME at the 1968 Materials Engineering Congress. A further session, on the practical applications of superplasticity, is scheduled for the 1969 Materials Engineering Congress, at Philadelphia.

Until recently, the bulk of the papers published on superplasticity have been devoted to the theoretical considerations of the deformation mechanisms, with little about technical applications or use. However, with a clearer understanding of the phenomena and the development of a number of commercial superplastic alloys(7,8,9), the reporting of an increasing number of commercial applications may be anticipated.

Superplasticity in metals, because of its rather loose definition, can be divided into two distinct types, depending on the method used to produce the remarkable extension. The first type, which depends upon the structure of the metal, more closely resembles the action of hot glass in that the deformation may occur at a steady temperature which is always greater than 0.4 of the melting point of the metal in degrees Kelvin(6) ($>0.4T_M$). An example of this type of superplastic deformation is presented in Figure 1, in which tensile bars of 85% Zn-14% Al-0.7% Cu alloy are shown before and after straining at 250°C (482°F); elongation was 980%.

The second type is sometimes known as Phase Transformation Plasticity and is produced by cycling the stressed metal many times through an elevated-temperature phase transformation. Using the cyclic method, DeJong and Rathenau(10), Clinard and Sherby(11), and Oelschlagel and Weiss(12) have been able to produce unusual deformations at low stress in iron and have shown it to be abnormally weak in the region of the transformation. Weiss(13) has produced an elongation of 750% in a steel by cycling it 255 times under load through the α-γ transformation. An example of a tensile specimen deformed in this manner is presented in Figure 2. Lozinsky(14) has shown that a double-necking effect occurs in Armco iron, titanium, and zirconium, on thermal cycling under load, while Kot and Weiss(15) have shown that the strain for fast temperature cycling (about 1 minute per cycle) through the α-β allotropic phase boundary for titanium exceeds to a considerable extent the strain due to normal creep without transformation cycling. A similar effect was observed in the case of three plain carbon steels.

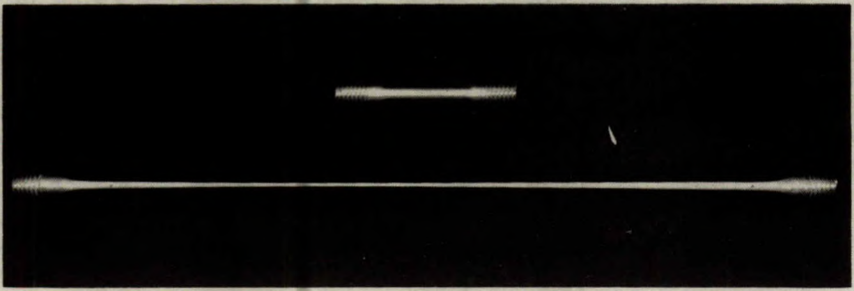
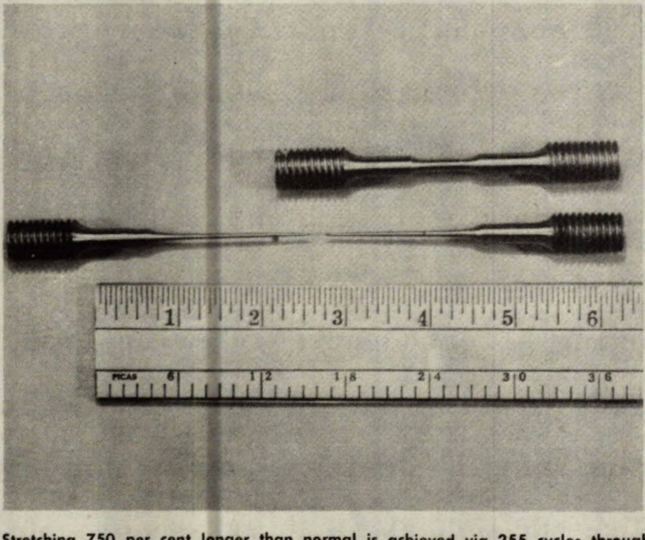


Figure 1. Superplastic Zn-Al alloy before and after deformation at 250°C (482°F), $\dot{\epsilon} = 0.5/\text{in.}$(44).



Stretching 750 per cent longer than normal is achieved via 255 cycles through steel's transformation temperature, claims Dr. V. Weiss, University of Syracuse

Figure 2. Cyclic or phase transformation plasticity produced this 750% elongation(13).

This cyclic method of producing abnormal elongations at low stress is of theoretical interest, but its scope of application appears to be limited. Discussion of this type of superplasticity involving thermal cycling will be terminated here, and further references to the superplastic phenomenon refer only to the first type described.

METALLURGICAL FACTORS ASSOCIATED WITH SUPERPLASTICITY

Some of the more important metallurgical factors required for a metal to exhibit superplastic properties have been identified and, although it is not possible to predict that under these conditions every metal would be superplastic, they do delineate some regions where superplasticity might be found.

The metal or alloy must have a fine grain structure, that should be less than 3 microns(16,17). A finer grain structure increases the effect and also increases the rate at which superplastic deformation may take place. Grain-boundary structure, grain shape and grain orientation are also of importance, as shown by Martin and Backofen(18) and Holt(19,20), but their range and limitation are not fully defined. Non-metallic inclusions or precipitates in the grain boundary tend to inhibit the effect, as do long elongated grains, while a random orientation of equiaxed grains is highly satisfactory.

Grain growth at the deformation temperature must be low, because of the limiting grain size factor for the phenomenon. Morrison(21) has demonstrated the results of grain growth in his experiments on low-alloy steels where superplasticity present in the initially fine-grained steel was rapidly lost as the grains grew at the deformation temperature.

Many superplastically deformed metals show little or no grain growth after deformation, nor in many cases do the grains appear to develop any directionality. This type of deformation is described by Packer and Sherby(22) for 78% Zn-22% Al alloy, and by Hayden, Gibson, Merrick and Brophy(23,24) for a 48% Ni, 39% Cr, 10% Fe alloy. The latter alloy elongated 1000% at 981°C (1800°F) at a rate of 0.2/min, the original 1-micron grain size was retained, relatively equiaxed, and the grains were nearly dislocation-free.

A duplex structure, wherein the two phases tend to limit their respective grain sizes because of the interference of the other phase, has been used as a method of limiting grain growth in the superplastic temperature range. Associating this with the very fine grain size of "micro" grains required for superplasticity, Gibson, Hayden and Brophy(25) coined the word "Microduplex" to describe the structure which is frequently associated with superplasticity. Superplastic alloys, however, do not have to have duplex structure. Thus, single-phase solid-solution alloys such as Sn-1% Bi(26), commercial purity zinc(27), titanium(28), pure nickel(29), and plutonium(30), all exhibit superplastic deformations.

The phenomenon does not appear to be restricted by the number of phases present, per se, but by their type and distribution(8) and the grain boundary conditions. Marder(31) attributes his failure to find superplasticity in a fine-grained ferrite-carbide structure to the fact that the structure was not equiaxed and that both phases were not ductile. Martin and Backofen(18) have shown that a mixture of lead and tin built up by electroplating thin layers of each element about 0.5 micron thick can be superplastic, but that when the two metals are co-deposited in a structure so fine as to be unresolvable the alloy is not superplastic.

Superplasticity has been associated with compositions and temperatures in the vicinity of regions where atomic rearrangement of atoms occurs (particularly phase changes in eutectic and eutectoid alloys), and with metastability in these regions(5,6). Ball and Hutchison(32) have shown that metastability of structure is not a requirement for superplastic behaviour in 77% Zn-23% Al alloy. Whether the phase change <u>per</u> <u>se</u>, rather than its effect in generating the required fine-grain structure at a propitious temperature, contributes to the superplasticity, is not entirely clear. However, transformation boundaries do designate areas where superplasticity is likely to be found.

The superplastic phenomenon has a temperature requirement; namely, the temperature of the metal at deformation should exceed $0.4T_M$. Thus, some alloys may deform superplastically at room temperature, e.g. as Sn-5% Bi whose room temperature equivalent is $0.6T_M$(33), while other high-melting-point alloys must be deformed at appropriately high temperatures to have the phenomenon operative. That the effect is not most pronounced immediately below the solidus is surmised to be due to the rapid grain growth that would occur in this region and thus destroy the effect.

Another restriction on the phenomenon concerns the rate of straining. In general, the superplastic effect is observed when the strain rate is between 10^{-4}/min and 10/min, which would include the range normally used in mechanical tensile tests of 0.003/min-0.5/min(34) but is considerably slower than the strain rate used in normal metal-forming operations, which are in the order of 1-9000/min(35). Thus, even in the Zn-Al eutectoid alloy, nominally the best superplastic performer, the rate of deformation is low in comparison with normal industrial rates. However, many researchers(6,16,20,33, 36,37 and 38) have indicated that decreasing the grain size and/or increasing the temperature serves to increase the strain rate at which superplasticity may be found. Schadler(38) indicates that in certain instances the strain rate $\dot{\epsilon}$ is proportional to $-\frac{1}{(L)^5}$

where L is the mean intercept grain diameter. Thus, a small decrease in grain size should produce a marked increase in strain rate. On this basis he predicts strain rates of 1/min for 0.5-micron grain size for an AISI 1340 steel. It would appear that to achieve more reasonable deformation rates, alloys must be developed with a finer grain size, as this is the predominant factor for producing superplasticity. The alloy's ability to retain its fine structure at superplastic-deformation temperatures should also be developed.

To the grain structure, temperature, and mechanical factors required for superplasticity, one further and major condition must be added; namely, that the metal have a strain rate sensitivity m (also known as strain rate exponent) greater than 0.3(16).

This factor is derived from the stress-strain relationship described by

$$\sigma = k \, \dot{\epsilon}^{m}$$

where σ = stress,

$\dot{\epsilon}$ = strain rate,

m = strain rate sensitivity or exponent,

k = constant.

This expression is commonly used to describe flow in viscous liquids where the stress is not a function of the strain, as is usual in metals, but of the strain rate. One of the characteristics of this type of flow is its uniform extension, even where small discontinuities in the fluid are present, and this quality permits some tolerance of the slightly weaker areas characteristically found in metals as a result of vacancies, dislocations, and inclusions. This tolerance reduces the tendency of a straining metal to "necking" and permits its extensive deformation superplastically. When the strain rate sensitivity m = 1 the expression reduces to

$$\sigma = k\,\dot{\epsilon}$$

which describes Newtonian viscous flow, with the stress varying directly with the strain rate.

Backofen and his colleagues(6) were the first to quantitatively associate superplasticity with resistance to neck formation and to correlate the latter with the strain-rate sensitivity, m, while Packer and Sherby(22) showed, by a simple geometric argument, that the higher the value of m the greater the resistance to neck formation.

When m = 1, the rate of diminution of area will be the same along the length of a specimen, regardless of any differences in cross-sectional area. Thus, the specimen after elongation would be an extended duplicate of the original specimen, including any notches and dimensional defects. The smaller areas support the same load as the larger areas; only the rates of deformation of the two are different. This is in contrast with the usual behaviour of ductile metals, where structural defects on even an atomic scale can reduce a cross-section area supporting the load, and this area strains, strainhardens, only to strain further at a higher load, until a neck develops. When strain hardening cannot keep up with the area reduction, then the metal fails in the necked region.

In the case of a superplastic metal, the strain-hardening effect is associated with the rate of deformation. Thus, the smaller cross-sectional areas would strain more rapidly, and consequently strain-harden more than the slower-straining, larger areas, which also must strain in order to harden and bear the increasing load. No necking occurs with this type of deformation when $m = 1$. When $m < 1$ the small cross-sectional areas will reduce more rapidly than the larger areas. As m drops in value from 1 this effect gradually becomes more pronounced, and $m = 0.3$ appears to be about the limit of the superplastic range. At $m = 0.2$ the metal behaves normally and necks with less than 100% elongation.

The strain-rate sensitivity m is usually calculated from the expression

$$m = \delta \log \sigma / \delta \log \dot{\epsilon} .$$

m is not constant but varies with the strain rate. In normal metals, m is less than 0.2, frequently less than 0.05, and it normally decreases slightly and steadily with increasing strain rate(17). In superplastic metals, m is usually in the range 0.3 to 0.8 and does not vary uniformly with strain rate but shows a peak in the m vs $\dot{\epsilon}$ curve. Backofen et al.(6) have plotted constant m values on strain-rate vs temperature coordinates that give an indication of the complex variations of m under the influence of these variables for a Zn-Al alloy. The resulting plot produces lines resembling contour lines on a map, describing several peaks.

Theoreticians have expended considerable effort to mathematically describe stress-strain relationships found in superplasticity. These equations and their inter-relationships become complex, as at least three mechanisms have been shown to be involved: Newtonian creep, Herring-Nabarro creep, and Coble creep. Zehr and Backofen(39) have suggested that the mechanism underlying the superplastic effects in Pb-Sn eutectic alloy and in ZK60 magnesium alloy is one of non-Newtonian grain-boundary sliding and diffusional creep acting in parallel, together with a more common non-Newtonian slip-creep acting in series.

Strain-rate sensitivity, m, is now commonly interpreted as indicating superplasticity in a metal when greater than 0.3. Thus, Holt(20), in his work on superplasticity in Al-Zn eutectoid alloy, was able to study the effect by compressive deformation rather than in tension. He employed two methods to generate the required stress and strain-rate data from which he calculated m. In one case, the cross-head velocity was varied in steps while noting the change in stress; in another, a series of tests was made at constant velocities to a fixed strain of -0.2. This variation is of interest in that the superplastic effect is shown to exist for metal deformed in compression as well as in tension. From the practical standpoint of testing alloys, such a method eliminates the requirement of a furnace with a long uniform zone of heating. Holt's results showed that the measurement of the m value alone is sufficient to establish superplasticity of an alloy.

Superplasticity might also be recognized by its curious property of showing no strain hardening of the metal and the persistence of equiaxed grains even after severe deformation(40,41,42 and 43), in contrast with other deformation processes, including creep.

Although the complete mechanism for superplasticity in metals is not fully understood to-day, it is generally accepted that the complex mechanism depends upon grain-boundary sliding by the climb-glide of dislocations in the boundary surfaces(40,43). A number of equations have been derived that are represented as describing the process, but as yet none has received general acceptance.

METAL OR ALLOY SYSTEMS DISPLAYING SUPERPLASTICITY, AND METHODS USED TO CONDITION THEM

General Considerations

A large number of alloys have been reported to have superplastic properties (see Table 1) and some of these will be briefly reviewed here. However, it should be remembered that the appreciation of some of the important factors contributing to the phenomenon, has only recently been recognized; thus, many other alloys may soon be developed that will show this property,

TABLE 1

Some Metals That Have Superplastic Properties

	Metal or Alloy System	Reference
1.	Ag-Cu, eutectic	54
2.	Al-Cu, 10-40% Cu, 89-90% Cu	8, 50, 51, 52, 53
3.	Al-Si	55
4.	Al-Zn, 0.2-40% Al	45, 2, 3, 6, 9, 20, 32, 37
5.	Bi-Sn, 1% Bi, 5% Bi	1, 26, 33
6.	Cd-Bi	56
7.	Cr-Co, 30% Co	57
8.	Cr-Ru, 24% Ru	58
9.	Cu-Ni	5
10.	Cu-Zn, 60-40 Brass	4, 53
11.	Mg-Al, eutectic	59, 60
12.	Mg-Cu	59
13.	Mg-Zn (Mg ZK60)	61
14.	Nickel pure	30
15.	Pb pure	62
16.	Pb-Cd	53
17.	Pb-Th	63
18.	β-Plutonium	64
19.	Sn-Pb	1, 18, 36, 39
20.	Sn-Sb	40
21.	Stainless alloy, 52% Ni, 39% Cr, 8% Fe	23, 24
22.	Stainless steel	7, 25
23.	Type 1300 carbon steel	13, 21, 38
24.	Titanium, commercial	28
25.	Ti-alloys, 6% Al, 4% V, 5% Al, 2.5% Sn	28
26.	W-Re	65
27.	Zirconium	66
28.	Zircalloy 4	28
29.	Zinc, commercial	27

as methods to produce the required fine grain size are developed. Many of the alloys reported are of little more than scientific interest, as they have been developed in order to help elucidate the mechanics of the phenomenon, not for structural or economic application. Thus, many of the alloys are made from low-melting-point or high-cost metals.

The best superplastic alloy appears to be the zinc-aluminum eutectoid (22% Al). Pollard(44) has shown that the Zn-14% Al alloy can be superplastic, while Cook and Risebrough(45) report superplasticity in an alloy containing only 0.2% Al. Holt has shown that Zn alloys containing up to 40% Al may be superplastic if mechanically worked.

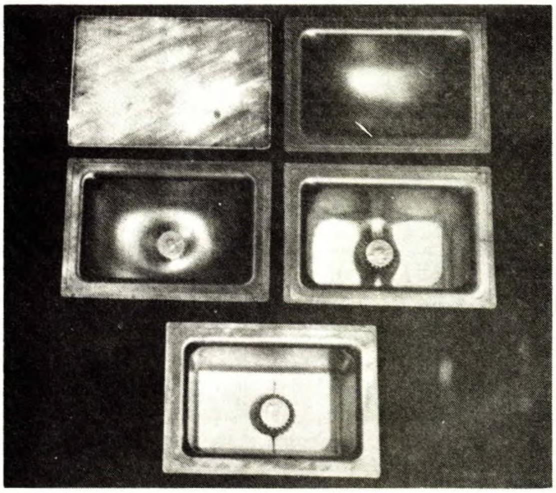
The eutectoid alloy has shown deformation of 1600% and performs with some reliability. It forms the basis for several commercial alloys(9,44). This alloy can develop the fine grain structure necessary for superplasticity by a single heat-treatment. The alloy is heated above 275°C (527°F) into the solid single-phase region for 50 hours. The alloy is then rapidly quenched into water or icy brine and allowed to regain room temperature. The single phase then decomposes spontaneously into a very fine two-phase eutectoid, which can have a grain size in the order of 0.5 micron. This reaction is accompanied by evolution of heat for a period of about 10 minutes as the single phase breaks down. The alloy is then in a condition for heating to the temperature at which superplastic deformation will be carried out. The most propitious temperature for this operation depends upon the rate of deformation employed. Backofen et al.(6) have shown that two peak values of m, the strain rate sensitivity, exist for this alloy that correspond to the best condition for superplasticity. One is in the neighbourhood of 250°C (480°F) with a strain rate of 1/min, while the other is above the transformation temperature at 300°C (572°F) with a strain rate of 10^{-2}/min. This alloy has been vacuum formed to great draw depths and shows excellent transfer of details (Figures 3 and 4) from the mould to the work piece.

Iron, low-alloy steels, stainless steels, and Ni-Cr-Fe alloys have all been demonstrated to have superplastic properties. These are not present to the same degree as found in the Zn-Al alloys, but extensions of greater than 960% for a 52% Ni, 39% Cr, 8% Fe, 1% Ti alloy have been recorded(23).

Superplastic stainless steels have been shown by Gibson, Hayden and Brophy(25) to have elongations of 600% at a strain rate of 0.266/min and in the temperature range 871-981°C (1600-1800°F). Such steels have a nominal composition of 25-26% Cr, 6-7% Ni, 0.02-0.045% C, 0.15-0.60% Ti, balance iron. In this type of alloy, it has been found that $Cr_{23}C_6$ will precipitate at the grain boundaries and inhibit superplasticity unless controlled. Titanium additions are made to precipitate TiC rather than $Cr_{23}C_6$, as the TiC precipitates randomly throughout the structure and does not limit its elongation. The steels must be conditioned into a microduplex structure by proper

Wire screen, plastic letters, check ability of superplastic metal to reproduce fine detail. Technique resembles plastic vacuum forming

Figure 3. Fine detail reproduced in Zn-22% Al eutectoid superplastic deformed by vacuum forming(13).



These are five blanks arranged to show the progress of forming in the die (above) over a period of perhaps 4 minutes

Figure 4. Superplastic Zn-22% Al alloy is hot vacuum formed into a deep sink. The hob in the middle is an IBM typewriter letter punch(13).

working and heat treatment to develop its superplastic properties(46). There are two methods of accomplishing this. The first is by heating to 1204-1259°C (2200-2300°F), in which condition the alloy is fully ferritic, and hot working from this temperature by rolling or forging while the temperature of the metal is allowed to fall into the two-phase, ferrite + austenite, region. This procedure causes the deformed large ferrite grains to simultaneously recrystallize and break down with the precipitation of the austenite phase along grain boundaries. Such an operation produces a 3.5-micron grain size structure of austenite grains in a ferrite matrix. Superplastic deformation of the steel follows at about 1700°F.

Another method which develops finer grains and consequently greater superplasticity consists of hot rolling from 1204°C (2200°F), cooling to room temperature, and cold rolling to 80% reduction, then reheating for an hour at 926°C (1700°F). This produces a 2.2-micron grain structure of austenite grains dispersed in a ferrite matrix. Superplastic elongations are obtained only at slow strain rates, about 0.3/min, but at the high strain rates of rolling and extrusion, part of the superplastic effect is still apparent in the slightly reduced stress required to perform these operations. The microduplexed stainless steel requires rolling forces and extrusion pressures similar to those of ferritic stainless steels Type 430 and considerably less than those of the austenitic Type 304 stainless steels. At room temperature, the microduplexed steels are superior in tensile strength to both ferritic and austenitic, high- and low-alloyed single-phase steels, while the ductility is as high or higher, fatigue strength is better, and corrosion properties are maintained. It would appear that these superior properties are as much responsible for the extensive research being carried out on the microduplexed steel as is interest in its superplastic properties(25,43,45,47 and 48).

The Ni-Cr-Fe alloys better demonstrate superplasticity in high-temperature metals, as the effect is more pronounced. Hayden, Gibson, Merrick and Brophy(23,24 and 46) have investigated these alloys, developed several procedures for producing the required microduplex structure, and showed that the process could be successfully scaled up to produce ingots of 10 tons. One of their methods consisted of heavily hot-working the cast billet as its temperature dropped from 1204°C (2200°F); another involved heating the alloy to 1204-1259°C (2200-2300°F) where the structure was single-phase, f.c.c. γ, quenching to room temperature, heavily cold-working, then reheating to 760-871°C (1400-1600°F) where recrystallization of the deformed f.c.c. γ occurred, together with the precipitation of b.c.c. α. The combined reactions produced the required fine structure. The cold-working step is necessary to supply the energy for recrystallization, without which the precipitation would occur largely as a second phase within a coarse-grained γ matrix. Such a structure would not be superplastic. Superplastic deformation was demonstrated at strain rates as high as 1/min at 926°C(1700°F) and 981°C(1800°F).

Carbon and Low-Alloy Steels

Schadler(38) has shown superplasticity in steels similar to AISI 1340, which may contain up to 2% Mn, by producing steel with an ultra-fine grain size of about 1 to 5 microns in a duplex structure. He strained tensile specimens of the steel at 10^{-2} to 10^{-3}/min below the γ transition temperature.

He found the strain-rate sensitivity, for this type of steel in the $\alpha + \gamma$ condition at 718-733°C (1325-1351°F), to be as high as 0.6 but that it decreased smoothly to 0.16 at higher strain rates. The steel in the austenitic condition at 782°C (1438°F) was not superplastic. The strain-rate-sensitivity was found to be 0.16 and was insensitive to strain-rate variations. Steels containing a moderate amount of manganese, for example AISI 1340, had higher strain-rate sensitivities than steels with low manganese contents. The carbon content of the steel was shown to have little effect on strain-rate sensitivity, thus his results appear consistent with those of Lee and Backofen(28) who reported $m \approx 0.25$ for Armco Iron.

Schadler produced his 1- to 5-micron grain size in 0.050-in.-thick sheet by a method described by Grange(49) for commercial steels. This consisted of up-quenching into a salt bath at 825°C (1517°F), a 20-second hold, then a quench into ice water for 10 seconds. This sequence was repeated 8 times to produce the required grain refinement. More cycling did not produce further grain refinement but with fewer cycles than 8 the structure was coarser.

He concludes that, for steel with a 1- to 2-micron grain size, commercial forming operations would only be marginally useful, because of the low strain rate. However, if the grain size could be reduced to 0.5 micron, superplastic steel could be fabricated at present-day working strain rates.

Schadler has also shown that these steels can be superplastic when the microduplex structure is $\alpha + Fe_3C$ at temperatures of 630°C (1166°F) or 660°C (1220°F) but at low strain rates. The strain-rate sensitivity is comparable with that found in the $\alpha + \gamma$ region, namely, $m = 0.6$.

Morrison(21) has also recently investigated superplasticity in a number of low-alloy manganese AISI Type 1300 steels in the ferrite + austenite regions. He micrograined his two-phase steels by hot-rolling at 900°C from 1 in. to 0.15 in. in several passes. (At this temperature the steel consisted of approximately 50:50 ferrite-austenite grains.) On air-cooling, the metal had a grain size of about 2 microns. In an effort to stabilize the grain size to temperatures above 800°C (1472°F), the practical limit for C-Mn steels, small alloy additions of ferrite stabilizers Al, P, or Si were made. This permitted the temperature to be increased to 900°C (1652°F), but did not prevent grain growth. These steels displayed some superplasticity, with m values as high as 0.5 in the 800°C (1472°F) to 900°C (1652°F) range for strain rates of the order of 10^{-2}/min. The strain-rate sensitivity was found to decrease during the deformation, accompanied by a decrease in ductility. These effects were attributed to the grain growth. Superplasticity was not observed at strain rates of commercial interest.

Copper Alloys

Two types of alloys in the Al-Cu system, aluminum-rich and copper-rich, have been shown to have superplastic properties. Presnyakov and Chervyakova(50) have reported superplasticity in the cast eutectic, 33% Cu. Petty(51) has shown that not only the eutectic alloy composition, but also a whole range of alloys from 8% Cu to 40% Cu when strained at 6×10^{-3}/min above 400°C (752°F), has large elongations when made from hot extruded wire. Holt and Backofen(52) reported superplasticity, with strain-rate sensitivities as high as 0.6 for the hot-worked eutectic alloy, at 520°C (968°F) with a strain rate of 6×10^{-1}/min. This material had a grain size of about 2 microns. The alloy remained superplastic as the grain size increased to as much as 7.7 microns, but only because of appropriate reduction in the strain rate.

Alloys at the high copper side of the Al-Cu system are also reported to be superplastic. Stamford(53) reported that a eutectoid aluminum bronze containing 11.8% Al, properly conditioned and strained at 540°C (1004°F), can have an elongation of 535%. The technique used to produce the required fine grain size consisted of quenching the eutectoid alloy from the all-β condition to produce an $\alpha + \gamma_2$ structure in a martensitic-type transformation, which was then further broken up by extrusion at 500°C (932°F). The final grain size was less than 5 microns.

A commercial alloy(8) containing 85% Cu, 10% Al, 4.5% Fe, with limits on Zn and Pb, has been reported to have superplastic properties at 583°C (1081°F) when strained at 0.1/min. The conditioning treatment is not reported but it would appear to be similar to that used for the martensitic transformation discussed above. At room temperature this alloy is reported to have properties superior to phosphor-bronze or Cu 0.8% Be alloy.

Copper-zinc brasses have been reported to be superplastic, by both the Russians(4) and the British(53). Stamford(53) has elongated a 60-40 brass 500% at 500°C (932°F) at the optimum strain rate. His conditioning treatment consisted of quenching the alloy from the β phase, followed by hot extrusion at 500°C (932°F), which produced a fine-grain β structure containing a fine dispersion of α. This alloy loses its superplastic properties rapidly at 500°C (932°F) because of grain growth which limits its extension.

Other Systems

A number of other alloy systems, most of which have been listed in Table 1, have been reported to possess superplastic properties. It is anticipated that such a list will be vastly extended in the near future as present knowledge of the phenomenon's requirements become more widespread and other systems properly conditioned are appropriately tested.

SUPERPLASTIC VOID FORMATION

The generation of voids in superplastically deformed metal has been reported(6,16,22,25,44 and 53). In some systems, and with some investigators, the occurrence would seem to be almost routine, while with others it is a rarity or may not be mentioned. In normal metals, voids may be generated as the result of creep, or in the final stages of tensile testing (in the necked material), or, microscopically, where vacancies have condensed during deformation(67); these voids create a major weakness in the metal and can be an indication of imminent failure. In superplastic metals, however, under stress the occurrence of voids does not produce immediate overloading and failure; because of the metal's resistance to necking, the section tends to continue to bear its share of the load till the whole specimen is quite thin. The occurrence of voids usually reduces the superplastic elongation but does not eliminate it. Pollard(44) has shown photomicrographs of superplastically-deformed Zn-14% Al alloys in which many of the voids appear to be associated with the interface between the grains. He reports that, although the metal in the thinner sections of a test specimen looked porous, the specimen had drawn to a large extension before failure. There is an implication, from the reports of voids produced during superplastic deformation, that voids occur when some of the conditions for superplasticity are borderline.

Stamford(53) reports the extensive occurrence of voids in superplastic aluminum bronze (10% Al) even when it was deformed at the optimum strain rate at 540°C (1004°F). He found that the voids rendered the material unsuitable for subsequent commercial uses. He also reported the formation of voids in 60-40 brasses, leaded and non-leaded, at 500°C (932°F) but noted that grain growth was occurring at this temperature.

REAL AND POTENTIAL APPLICATIONS

Examples of the application of a superplastic metal to actual product fabrication are relatively rare at present. Backofen(6) demonstrated with the zinc-aluminum eutectoid alloy that superplastic sheet 0.030-in. thick could be blown up into a partial bubble at 260°C (500°F) with low pressures (30 psi nitrogen) when clamped to a die with a 4-in.-diameter hole. The bubble produced had a diameter of nearly five inches, expanding well beyond the diameter of the die hole. The superplastic deformation was slow, requiring, in this case, 20 hours.

Fields(68) showed how the same alloy could be hot-deformed by vacuum, in which case the sheet was drawn into a deep mould. Two examples of the final product are shown in Figures 3 and 4. A similar type of flat-bottomed basin, using a special laboratory apparatus that combines the application of vacuum, gas pressure and mechanical pressure, was formed with the same alloy, at the same temperature, from 0.025-in. sheet in 4 minutes. With sheet of 0.100-in. thickness, 34 minutes were required for the deformation. The ratio of original thickness to minimum final thickness was 4.6:1. The surface area of the sheet increased over 200%. It is important to note that the thinnest section occurred in the centre of the deformed area, rather than on the corners as is common with conventional drawn parts. Fields(69) reports that many different shapes have been formed with this machine, some with re-entrant features and some with a transfer of fine details difficult to achieve by any other technique.

A more practical application of the superplastic effect is reported(70,71) to be used in the U. S. A. for the production of discs or hubs for aircraft turbines. Sintered alloy blanks are slowly hot-press formed in a die to make the finished disc. The superplastic-alloy blanks are made by hot-pressing and sintering high-nickel, high-cobalt alloy powder with carbides in such a manner that the resulting metal alloy has a very fine grain structure. The grain structure is very stable, showing little grain growth even at high temperatures. However, this property, which contributes to the superplasticity of the alloy, also makes difficult the elimination of superplasticity after forming.

The superplastic property of this alloy is reported to have been discovered unexpectedly when the alloy was hot-worked, and the researchers, recognizing the effect, developed it into a successful production-forming procedure to produce the large complex jet-engine turbine hub in high-strength, high-alloy metal.

Two British companies show(72), as a demonstration of the formability of their superplastic Zn-Al eutectoid alloy, the inner section of a refrigerator door (usually made in plastic) completely formed in their metal alloy. The forming was carried out at 260°C (500°F), using a vacuum-forming technique similar to that used for plastics. A second application(9) is in automobile door panels, which are either vacuum-formed or low-pressure-gas formed, at 260°C (500°F). The time required for this deformation is not given, but strain rate is indicated as a major consideration in the fabrication. Because of the formability of this alloy, more complex panels can be formed that would replace several steel pressings. Costs can be lowered further by using cheaper, though specially designed, dies of ceramic, cement, cast iron or aluminum, made possible because of the low deformation pressures required. A property of this alloy also contributes to its use, namely its superior corrosion resistance as compared with steel. However, its stiffness is less than one-third that of steel and it must be about 40% thicker for equal stiffness. This deficiency can be overcome by incorporating easily-formed strengthener ribs. The commercial production of this part has not as yet become a reality, although samples have been made.

Jovane and Morelli(73) have investigated the application of the superplastic effect to the coining process and report that a high degree of coining may be obtained at lower pressures than would be the case for ordinary metals.

Thus, by using a suitable superplastic metal it should be possible to use lower-strength dies and lighter presses. Production rates would be lower, however, because of the lower strain rate required for superplasticity.

"Die-less" wire drawing is another application of superplasticity that is being developed. The method, according to Stamford(53), could be used to produce either wire or long-shaped sections from a large-cross-sectional-area bar of the same relative dimensions as the final reduced but elongated product. By taking advantage of the superplastic properties of the metal when hot, and their absence when below $0.4T_M$, a limited cross-sectional volume of the original bar is heated into the superplastic range by induction and strained at a uniform and controlled rate so that the alloy draws down evenly to a greatly reduced diameter before cooling to the point where deformation ceases at the strain rate employed. The induction heating coil is gradually moved back over the original bar until the whole bar has been deformed. Large single-pass reductions are claimed to be achieved and, with proper control of the moving induction coil, even tapered sections are thought to be possible. With further development this process might be useful for the production of rods and wires. Any unevenness could limit its application in critical areas, particularly in the small diameters where small variations in the diameter could not be tolerated. Final sizing might have to be done through a conventional die to establish the mechanical reliability.

Several papers on the practical application of superplasticity are soon to be presented at the 1969 Material Engineering Congress in Philadelphia(74). These include such titles as "Die-less Wire Drawing", "Superplastic Forming in Bulging Dies for Hollow-Ware Production", "Aerospace Applications of Superplastic Forming", "Microduplex Processing for Improved Hot Workability and Mechanical Properties", and "Elevated Temperature Tensile Properties of an Aluminum Bronze CDA-619".

These titles add little to our knowledge about new applications of the phenomenon, except in the case of Backofen's paper on "The Bulge Forming of Hollow-Ware" which would appear to be a practical extension from the unsupported gas-pressure forming technique he had previously demonstrated. Internally forming the pieces would allow the die to support the metal after it is formed to shape. It should also limit excessive deformation and possible rupture, and allow easy strain-rate control.

A final example of the application of the superplastic effect, appearing in the next paragraph, must be qualified by noting that only part of the effect is used, but the application appears to be one of major importance.

With the superplastic stainless steels and Ni-Cr-Fe alloys the extreme elongation possible is seldom demanded, because to achieve it the deformation rate must be very slow. It has been found(23,25), however, that part of the superplastic effect persists in these alloys at normal metal-working rates, namely the lower load pressure required when rolling or extruding.

Thus, superplastic alloys can be hot-worked at normal rates at stresses similar to ferritic stainless steels, and advantage is now being taken of this fact. The great interest in these alloys also derives from their good mechanical properties at room temperature; i.e., they exhibit improved yield and ultimate tensile strengths, along with improved toughness, with no increase in hardness.

POSSIBLE PROBLEMS

Utilization of the superplastic properties of an alloy in many forming operations will seldom be as simple a procedure as is sometimes stated. Some of the difficulties that might be encountered are listed below:

1. Strain Rate - The strain rate for superplastic deformation is relatively slow, and must be maintained within limits - a particularly difficult task in multiaxial deformation.
2. Time - Deformations take time and cannot be speeded up with the application of more power, as in many operations, because of the nature of this process. The long times required add significantly to the cost of the operation.
3. Temperature - Most alloys must be heated to and maintained in the correct temperature range during the deformation. This may require heated dies or a furnace large enough to accommodate and release the formed product.

4. Corrosion - The elevated temperature and long exposure time can create an oxidation problem. Atmospheric control may be necessary.
5. Stress Requirements - These are usually low after deformation has started. Initial stress requirements, however, may be considerably higher.
6. Reliability - The metal must be properly pre-structured by several closely-controlled steps.

DEFORMATION OF PLASTICS

In contrast with the forming of superplastic metals in plastic-forming dies, a recent report(75) indicates that plastics are being cold-formed with machines previously used to form metals. In view of the fact that some of these plastics can have high strain-rate sensitivity and show a superplastic effect, we should consider the observations made concerning them relative to the behaviour of superplastic metals. It has been found that the usual tensile tests, being uniaxial and done at slow speed, do not give sufficient information on which to assess deep-drawing qualities of a plastic in which the deformation is multiaxial. To be deep-drawable in conventional metal-working presses, a plastic must be able to deform over a wide range of speeds and in several directions. ABS plastic (acrylonitrile-butadiene-styrene), although it deforms only about 30% in a normal tensile test, does so over a wide range of deformation speeds, and so is compatible with the drawing speeds used in most metal-drawing presses in contrast with the plastic nylon, which can be strained 300% at 0.2/min but which will strain only 10% at press speeds of 2000/min. This latter plastic is not considered satisfactory for deep drawing.

Carrying these findings over into the field of superplastic metals, it would appear that nylon with its superplasticity at low deformation speeds would relate more closely with superplastic metals than ABS plastic. However, ABS has been found superior to nylon for deep drawing on conventional-metal deep-drawing equipment, despite nylon's higher ultimate strain and higher strain-rate sensitivity. Thus, we might infer that superplastic metal would not prove particularly beneficial or even satisfactory in conventional drawing equipment.

This conclusion appears to be borne out by the lack of published reports of satisfactory deep-drawn articles being formed on such readily available equipment. There are numerous reports of excellent deep-drawn articles being formed from superplastic metals but using specialized equipment at low strain rates and low stress levels. However, the rate of deformation used is usually at least an order of magnitude slower than in conventional metal-working equipment.

In our present industrial atmosphere such a loss of production would normally defeat such a process even if material costs were equal. To be attractive industrially, the superplastic process, because its deformation rate cannot be speeded up, must be applied to a special category of products rather than compete with commonly drawn or stamped articles.

DISCUSSION OF THE PLACE OF SUPERPLASTICITY IN METAL FORMING

The requirements of the superplastic deformation process are so numerous and restrictive as to limit its application to metal-forming operations which can tolerate a longer forming time and the additional prior conditioning and heating. The process cannot compete directly with normal metal stamping and drawing operations with their rapid high-volume, low-cost production. The superplastic process, being metallurgically more sophisticated and demanding, will be applicable in areas where normal processes are inefficient and expensive. The forming of large pieces could be done superplastically at low stress with heated dies that require no great strength. The energy requirements would be low even for the deformation of thick sheets, and could be supplied by the weight of the die itself sinking closed, possibly supplemented by addition of a removable static weight to initiate the operation. The rate of deformation would be controlled by the stress on the bearing areas. An automated program of loading and temperature control with deformation might be required for high-efficiency production. The articles produced need not be a plain section but could contain deep indentations and ridges. Application to such items as the large sidewalls for modern unitized transport containers, the exteriorly hung building panelling, and even whole box-car sides with reinforcing ribs and corners integrally formed, would appear to be practical areas for development for the lower-temperature alloys, such as the Zn-Al eutectoid.

This process would also be suitable for deformations of large parts made from high-strength, high-alloy metals where normal fabrication methods would require extremely heavy tooling and pressures.

The superplastic process, because of its great formability, makes it possible to form a more complex single part to fulfil the function of a number of parts that must be assembled, as for the automobile door panel previously mentioned. The use of the method in such a case would appear to be marginal unless other advantageous factors were present. The use of the bulge-forming method would appear to be much more favourable and adaptable to superplastic deformation. The deforming medium, whether gas or fluid, can be readily controlled to continuously provide the optimum strain rate for deforming the metal. By expanding the metal against the die, the formed metal is supported and close tolerance pieces of intricate design can be produced. Industrially, bulge forming is now being used to deform copper and steel parts up to 12 inches in diameter(76). Using superplastic materials, production of even larger pieces with weaker and cheaper dies could be anticipated.

RECOMMENDATIONS

Knowledge of the requirements for superplasticity is now in the process of dissemination amongst the metallurgical community, although theoretical metallurgists may argue for years about the specific mechanism of the process . The effect of the information will be an appreciation of the deformation benefits to be derived from a fine grain size at intermediate temperatures and a new look at the properties of fine-grained polyphased alloys of many metals that have been neglected for years.

Following this line should lead to discovery of several new superplastic systems and, hopefully, some alloys with improved properties. Much of the practical usefulness of superplastic alloys today comes from the improved properties of the alloys themselves and not from the fact that they are superplastic. A superplastic alloy must have its other properties at least comparable with normal alloys when put into service, or it will not prove a success. But it is interesting to note that at least two superplastic alloys, stainless steel and aluminum bronze, have better properties than the alloys they were derived from.

Practical applications of superplasticity on anything but a small scale areprobably several years away. As the operations are scaled up to form larger parts, the more applicable and successful the process should become. Already the Zn-Al eutectoid alloy has been continuously cast on a Hazelett machine and 5-ft-wide sheet rolled in England. Some development on large dies and control systems required to form big parts would be useful for such Canadian industry as might wish to adopt this method of production.

Die-less drawing is also in its infancy, and such a project might have wide application if it could be successfully developed.

The use of the extrusion press to break down the grain structure of many alloys to a suitable size for superplasticity suggests a multiple-chambered extrusion die for extruding complex or difficult shapes. The alloy would be grain-refined in passing into the first chamber, and, now fine grained and superplastic, it would pass easily and at low pressure through the second finishing orifice. Such a radical die design might be tried.

The principles of superplasticity might also be applied to difficult-to-form metals and alloys, such as beryllium, with some prospect of success.

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REFERENCES

1. C. E. Pearson, "The Viscous Properties of Extruded Eutectic Alloys of Lead-Tin and Bismuth-Tin", J. Inst. Metals 54, 1934, 111.

2. A. A. Bochvar and Z. A. Sviderskaia, "Superplasticity Phenomena in Zinc-Aluminum Alloys", Izv. Akad. Nauk SSSR, Otdel. Tekh. Nauk 9, 1945, 821.

3. A. A. Presnyakov and V. V. Chervyakova, "On the Subject of 'Super-Ductility' of Alloys", Physics of Metals and Metallography (Pergamon Press Translation), 8(1), 1959, 114.

4. A. A. Presnyakov and G. V. Starikova, "The Anomaly in the Ductility of Two Phase Brasses", Physics of Metals and Metallography (Pergamon Press Translation), 12(6), 1962, 84.

5. E. E. Underwood, "A Review of Superplasticity", J. of Metals 14, 1962, 914.

6. W. A. Backofen, I. R. Turner and D. H. Avery, "Superplasticity in an Aluminum-Zinc Alloy", ASM Trans. Quart. 57, 1964, 980.

7. Anon., "New Stainless Steel Forms Easily into Stron Shapes", Tool Mfg. Engineering 59(6), Dec. 1967, 28.

8. Anon., "Superplastic Aluminum Bronze: Stronger - But Cheaper - Than Phosphor Bronzes", Materials Engineering 67, 1968, 32.

9. Anon., "Prestal Described", Metal Bulletin, Nov. 1, 1968, 26.

10. M. DeJong and G. W. Rathenau, "Mechanical Properties of Iron and Some Iron Alloys While Undergoing Allotropic Transformation", Acta Met. 7, 1959, 246; Acta Met. 9, 1961, 714.

11. F. W. Clinard and O. D. Sherby, "Strength of Iron During Allotropic Transformation", Acta Met. 12, 1964, 911.

12. D. Oelschlagel and V. Weiss, "Superplasticity in Steels During the Ferrite ⇄ Austenite Transformation", Trans. ASM 59, 1966, 143.

13. Materials Report, "Superplasticity: Metals Draw Almost as Easily as Hot Plastic", Steel 14, June 1965, 66.

14. M. G. Lozinsky, "Grain Boundary Adsorption and "Superhigh Plasticity"", Acta Met. 9, 1961, 689.

15. R. Kot and V. Weiss, "Transformation Plasticity of Steels and Titanium in Torsion", Trans. ASM 60, 1967, 566.

16. D. H. Avery and W. A. Backofen, "A Structural Basis for Superplasticity", ASM Trans. Quart. 58, 1965, 554.

17. Hurmuze Naziri and Roger Pearce, "A Guide to Superplasticity", Sheet Metal Industries 46, 1969, 11.

18. P. J. Martin and W. A. Backofen, "Superplasticity in Electroplated Composites of Lead and Tin", ASM Trans. Quart. 60, 1967, 352.

19. David L. Holt, "The Metallography of Superplasticity in Some Aluminum-Base Alloys", ASM Trans. Quart. 60, 1967, 564.

20. David L. Holt, "The Relation Between Superplasticity and Grain Boundary Shear in Aluminum-Zinc Eutectoid Alloy", TMS, AIME, 242, 1968, 25.

21. W. B. Morrison, "Superplasticity in Low Alloy Steels", ASM Trans. Quart. 61, 1968, 423.

22. C. M. Packer and O. D. Sherby, "An Interpretation of the Superplasticity Phenomenon in Two-Phase Alloys", ASM Trans. Quart. 60, 1967, 21.

23. R. C. Gibson, H. W. Hayden, H. F. Merrick and J. H. Brophy, "Superplasticity in the Ni-Fe-Cr System", Trans. ASM 60, 1967, 3.

24. H. W. Hayden and J. H. Brophy, "Interrelation of Grain-Size and Superplastic-Deformation of Ni-Cr-Fe Alloys", Trans. ASM 61, 1968, 542.

25. R. C. Gibson, H. W. Hayden and J. H. Brophy, "Properties of a Stainless Steel with Microduplex Structure", Trans. Quart. ASM 61, 1968, 3.

26. R. H. Alden, "Superplastic Behaviour of a Solid Solution of Sn-1% Bi Alloy", TMS, AIME, 236, 1966, 1633.

27. D.A.C. Williams, H. Naziri and R. Pearce, "Extended Plasticity in Commercial Purity Zinc", Scripta Metallurgica - to be published.

28. D. Lee and W. A. Backofen, "Superplasticity in Some Ti and Zr Alloys", TMS, AIME, 239, 1967, 1034.

29. S. Floreen, "Superplasticity in Pure Nickel", Scripta Metallurgica 1, 1967, 19.

30. S. D. Dahlgren and R. D. Nelson, "Grain Size Dependency of β-Pu Plasticity", J. of Metals 20, 1968, 78A.

31. A. R. Marder, "The Effect of Carbon Content Test Temperature and Strain-Rate on Superplasticity in Finely Divided Fe-C Alloys", TMS, AIME, 245, 1969, 1337.

32. A. Ball and M. M. Hutchison, "Superplasticity in Aluminum Zinc Eutectoid", Metal Sci. J. 3, 1969, 1.

33. T. H. Alden, "The Origin of Superplasticity in the Sn-5% Bi Alloy", Acta Met. 15, 1967, 469.

34. ASTM Standards, Part 31, 1968, p. 14, p. 829.

35. Private communication, J. A. Perry, Dept. of Energy, Mines and Resources, Ottawa, Canada.

36. H. E. Cline and T. H. Alden, "Rate Sensitive Deformation in Sn-Pb Alloys", TMS, AIME, 239, 1967, 710.

37. T. Alden and H. Schadler, "The Influence of Structure on the Flow Stress-Strain Rate Behaviour of Zn-Al Alloys", TMS, AIME, 242, 1968, 825.

38. H. Schadler, "The Stress-Strain Rate Behaviour of a Manganese Steel in the Temperature Range of the Ferrite-Austenite Transformation", TMS, AIME, 242, 1968, 1281.

39. S. W. Zehr and W. A. Backofen, "Superplasticity in Lead-Tin Alloys", Trans. Quart. ASM 61(2), 1968, 300.

40. T. H. Alden, paper presented to CIM Conference of Metallurgists, Aug. 25, 1968, Vancouver, B. C.

41. T. H. Alden, "Grain Boundary Sliding as a Mechanism for Superplasticity", J. of Metals 20, Aug. 1968, 39A.

42. T. H. Alden, "Strain Hardening and Recovery in Superplastic Pb-5% Cd", Trans. Quart. ASM 61, 1968, 559.

43. H. W. Hayden, "The Effect of Microstructure on the Superplastic Behaviour of Fe-Ni-Cr Alloy", J. of Metals 20, Aug. 1968, 39A.

44. W. A. Pollard, "Assessment of Zn-14% Al Extrusion Alloy", Internal Report PM-R-68-19, Physical Metallurgy Division, Mines Branch, Dept. of Energy, Mines and Resources, Ottawa, Canada, 1968.

45. R. C. Cook and N. R. Risebrough, "Superplasticity in a Dilute Zinc Aluminum Alloy", Scripta Met. 2(9), 1968, 487.

46. H. W. Hayden, R. C. Gibson and J. H. Brophy, "Superplastic Metals", Sci. Amer. 220-3, Mar. 1969, 28.

47. H. W. Hayden and S. Floreen, "The Deformation and Fracture of Stainless Steels having Microduplex Structures", Trans. ASM 61, 1968, 474.

48. S. Floreen and H. W. Hayden, "The Influence of Austenite and Ferrite on the Mechanical Properties of Two Phase Stainless Steels having Microduplex Structure", Trans. ASM 61, 1968, 489.

49. R. A. Grange, "Strengthening Steel by Austenite Grain Refinement", Trans. ASM 59, 1966, 26.

50. A. A. Presnyakov and V. V. Chervyakova, "Superplasticity in the System Al-Cu", Izv. Akad. Nauk, Met i Topl 1958, [Tekhn], (3), 120.

51. E. R. Petty, "The Deformation Behaviour of Some Aluminum Alloys Containing Intermetallic Compounds", J. Inst. Metals 91, 1963, 274.

52. D. L. Holt and W. A. Backofen, "Superplasticity in Al-Cu Eutectic Alloy", Trans. ASM 59, 1966, 755.

53. M. S. Stamford, "Superplasticity", Copper 3, July 1969, 10.

54. H. E. Cline and D. Lee, "Strengthening Mechanisms of Directionally Solidified Fine-Grained Superplastic Ag-Cu Eutectic", J. of Metals 21, 1969, 84A.

55. A. A. Presnyakov and G. V. Starikova, "Condition Concerning the Appearance of Superplasticity in Cast Eutectics", Izv. Akad. Nauk SSSR, Otdel. Tekh. Nauk, 1, 1959, 75.

56. R. B. Nicholson, "The Relation of Microstructure to the Properties of Superplastic Alloys", J. of Metals 20, 1968, 39A.

57. J. R. Stephens and W. D. Klopp, "Superplasticity and Low Temperature Ductility in a Cr-30 At. Pct. Co Alloy", TMS, AIME, 236, 1966, 1637.

58. J. R. Stephens and W. D. Klopp, "Enhanced Ductility in Binary Chromium Alloys", TMS, AIME, 242, 1968, 1837.

59. A. A. Presnyakov and G. V. Starikova, "Metastability of Cast Eutectics Assessed in Connection with the Super-Ductility Effect", Physics of Metals and Metallography (Pergamon Press Transl.), 13 (5), 1962, 769.

60. D. Lee, "The Nature of Superplastic Deformation in Mg-Al Alloy Eutectic", J. of Metals 20, 1968, 56A.

61. W. A. Backofen, G. S. Murty and S. W. Zehr, "Evidence for Diffusional Creep with Low Strain Rate Sensitivity", TMS, AIME, 242, 1968, 329.

62. R. C. Gifkin, "The Stress Sensitivity of Lead at Low Stresses", TMS, AIME 239, 1967, 911.

63. R. C. Gifkin, "Superplasticity During Creep in Pb-Th Alloys", J. Inst. Met. 95, 1967, 373.

64. S. D. Dahlgren, "Superplasticity in Unalloyed β Plutonium", TMS, AIME 242(1), 1968, 126.

65. W. D. Klopp, W. R. Witzke and P. L. Raffo, "Mechanical Properties of Dilute Tungsten-Rhenium Alloys", NASA, TN, D3483, 1966.

66. R. W. Guard, J. H. Keeler and S. Reiter, "Observations on Elevated-Temperature Tensile Deformation", J. Metals 200, 1954, 226.

67. J. O. Stiegler, K. Farrel, B. T. Loh and H. E. McCoy, "Nature of Creep Cavities in Tungsten", Trans. ASM 60, 1967, 494.

68. D. S. Fields, Jr., "Thermo-Forming of Superplastic Alloy Sheet", Engineer's Journal 26(7), 1965, 97.

69. D. S. Fields, Jr., "Practical Applications of a Superplastic Alloy", Trans. ASM 60, 1967, 566.

70. J. C. Freshe and R. W. Hall, "Dispersion Strengthening"; in NASA Tech. Memorandum, Progress in NASA 1968 Program, p. 4.

71. Private communication (1968).

72. Anon., "A New Alloy, Prestal", Sheet Metal Industries 45(500), 1968, 904.

73. F. Jovane and P. Morelli, "On the Coining Process of Superplastic Alloys", J. of Metals 20, 1968, 58A.

74. Anon., "Practical Application of Superplasticity", ASM News Quart., Summer 1969, 20.

75. W. R. Meadors, "Metalworking Machines Form Plastics", Iron Age 202, Oct. 24, 1968, 69.

76. Takaski Ogura and Terumori Ueda, "Liquid Bulge Forming", Metalworking Production 112(17), 1968, 73.

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