Superplasticity
of Alloys, Intermetallides
and Ceramics
This book deals with the problems of superplastic deformation of materials. First of all, it is of interest due to its practical emphasis and the extensive treatment of superplasticity in alloys, intermetallics and ceramics. A significant conclusion which can be made by the reader is that this phenomenon bears the universal character and can be observed in virtually any crystalline material.

Much attention has been paid to the physical nature of the phenomenon. Various deformation processes operating in superplastic flow, i.e. grain boundary sliding, dislocation slip and diffusion creep have been experimentally studied.

A new point which is important for the theory of the superplastic effect is the establishment of interaction between intragranular slip and grain boundary sliding in superplastic deformation processes.

The present book substantiates the assumption that interaction between lattice dislocations and grain boundaries is significant in explaining the dominant contribution of sliding along grain boundaries during superplastic deformation. On the basis of the latest concepts in the physics of high-angle boundaries, it is shown that dislocations enter the grain boundaries and adopt a non-equilibrium structure under certain temperature-rate conditions typical of superplasticity. As a result, grain boundary sliding and migration are stimulated.

The main feature distinguishing this book from others is that superplastic deformation is considered, for the first time, not only as a phenomenon enabling large deformations to be produced without fracture, but as a new kind of materials processing permitting one to fix the fine-grained and homogeneous microstructures in the materials. This enables one to produce products not only of intricate shape but with good serviceability as well.

Dynamic recrystallization features allowing one to form micro- and submicron-crystalline structures in materials are also considered. It is shown that this processing enables one to refine the microstructure of virtually any kind of material and to transfer it into the superplastic state.

The main advantage of the present book is its in-depth consideration of the nature of the phenomenon and its exposition of the theoretical basis of the technology relevant to putting the results into practice. Commercial methods of refining the microstructure of materials are described. Superplastic deformation modes are given for the specific materials. The effect of superplastic processing on the properties of materials is evaluated. These concepts and methods can be useful in solving the problem of increasing the ductility of low-ductility materials.
In spite of the fact that this book presents data compiled from many studies, it is not a review of the literature but rather the result of the author's experimental and theoretical investigations. The author and his colleagues hope that this book will be of value both as an aid to learning and from a practical point of view.

Ufa, September 1992

Oscar A. Kaibyshev
## Contents

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Introduction</td>
<td>1</td>
</tr>
<tr>
<td>1. Structural Superplasticity</td>
<td>4</td>
</tr>
<tr>
<td>1.1 Phenomenology and Influence of the Initial Microstructure</td>
<td>4</td>
</tr>
<tr>
<td>1.2 Structure Evolution and Fracture Modes</td>
<td>21</td>
</tr>
<tr>
<td>2. The Nature of Superplastic Flow</td>
<td>30</td>
</tr>
<tr>
<td>2.1 Experimental Investigations of Superplastic Deformation</td>
<td>30</td>
</tr>
<tr>
<td>2.1.1 Grain Boundary Sliding</td>
<td>31</td>
</tr>
<tr>
<td>2.1.2 Intragranular Deformation</td>
<td>40</td>
</tr>
<tr>
<td>2.1.3 Diffusion Creep</td>
<td>52</td>
</tr>
<tr>
<td>2.1.4 The Interrelationship and the Role of Different Deformation</td>
<td>58</td>
</tr>
<tr>
<td>Mechanisms in Superplastic Flow</td>
<td></td>
</tr>
<tr>
<td>2.2 Micromechanisms and the Theory of Superplastic Deformation</td>
<td>61</td>
</tr>
<tr>
<td>2.2.1 Physical Models</td>
<td>62</td>
</tr>
<tr>
<td>2.2.2 Grain Boundaries in the Deformation Processes</td>
<td>67</td>
</tr>
<tr>
<td>2.2.3 A New Model of Structural Superplasticity</td>
<td>76</td>
</tr>
<tr>
<td>3. Microcrystalline Materials and Microstructure Refining</td>
<td>92</td>
</tr>
<tr>
<td>3.1 Structure and Properties of Microcrystalline Materials</td>
<td>92</td>
</tr>
<tr>
<td>3.2 Metallurgical Methods of Refinement</td>
<td>99</td>
</tr>
<tr>
<td>3.3 Heat Treatment</td>
<td>102</td>
</tr>
<tr>
<td>4. Superplastic Deformation of Magnesium Alloys</td>
<td>117</td>
</tr>
<tr>
<td>4.1 Effect of Chemical Composition, Structure, and Temperature-Rate</td>
<td>118</td>
</tr>
<tr>
<td>Conditions of Deformation on Ductility</td>
<td></td>
</tr>
<tr>
<td>4.2 Refining of the Magnesium Alloy Structure</td>
<td>126</td>
</tr>
<tr>
<td>4.3 Effect of Superplastic Deformation on the Properties of the Alloys</td>
<td>133</td>
</tr>
<tr>
<td>5. Superplasticity of Commercial Aluminium Alloys</td>
<td>148</td>
</tr>
<tr>
<td>5.1 Effect of Structure, Conditions of Deformation, and Chemical</td>
<td>149</td>
</tr>
<tr>
<td>Composition on the Superplasticity of Aluminium Alloys</td>
<td></td>
</tr>
</tbody>
</table>
5.1.1 Effect of Structure and Conditions of Deformation.
   Strain Rate .......................................................... 150
5.1.2 Effect of Chemical Composition ............................ 154
5.2 Processing Methods for Obtaining Microcrystalline Structure
   in Commercial Aluminium Alloys ................................. 160
5.3 Effect of Superplastic Deformation on the Structure
   and Properties of Aluminium Alloys ............................. 164
5.4 Effect of Grain Size on the Structural Strength
   of Aluminium Alloys ............................................... 171

6. Superplasticity of Titanium Alloys ............................... 175
   6.1 Plasticity and Superplasticity of the Titanium Alloys
       in the Single-Phase Region .................................... 176
   6.2 Superplasticity of Titanium Alloys in the Two-Phase Region . 188
   6.3 Methods of Obtaining Microcrystalline Structure
       in Titanium Alloys ............................................. 199
   6.4 The Effect of Superplastic Deformation
       on the Mechanical Properties of the Titanium Alloys .... 202

7. Structural Superplasticity of Steels ............................ 210
   7.1 Superplasticity of Iron, Plain Carbon, and Alloyed Steels .... 210
   7.2 Preparation of Fine-Grained Microstructure in Steels ........ 221
   7.3 Effect of Superplastic Deformation on Mechanical Properties .... 225

8. Superplasticity of Nickel-based Superalloys ...................... 231
   8.1 Effect of Structure, Deformation Temperature
       and Strain Rate on the Ductility of Alloys .................. 233
   8.2 Methods for Converting Alloys into a Superplastic State .... 243
   8.3 High-Temperature Properties of Alloys
       After Processing in the Superplastic Condition ............ 251

9. Superplasticity of Intermetallic Compounds ....................... 256
   9.1 Superplasticity of Alloys with Both Intermetallic
       and Metallic Phases .......................................... 258
   9.2 Superplasticity of Intermetallic Compounds ................. 261
   9.3 Refining of Microstructure in Intermetallics ............... 268
   9.4 Effect of Superplastic Treatment
       on the Physico-mechanical Properties of Intermetallics .... 272
       9.4.1 Mechanical Properties
           Under Ductile-Brittle Transition ........................ 272
       9.4.2 Mechanical Properties at Room Temperature ............ 275
       9.4.3 Magnetic Properties .................................... 277
List of Abbreviations and Symbols

CSL - coincident site lattice
DC - diffusion creep
GBD - grain boundary dislocation
GBS - grain boundary sliding
hcp - hexagonal close-packed (lattice)
HTMT - high-temperature thermomechanical treatment
IDS - intragranular dislocation slip
PFZ - precipitate-free zones
SP - superplasticity (superplastic)
SPDHT - superplastic deformation-cum-heat treatment
SPDT - superplastic deformation treatment
TLD - trapped lattice dislocations

\[ m = \frac{d \log \sigma}{d \log \dot{\varepsilon}} \] - coefficient of strain-rate sensitivity of the flow stress

\( \sigma \) - flow stress
\( \sigma_u \) - ultimate tensile strength
\( \sigma_{0.2} \) - offset yield stress
\( \varepsilon \) - strain
\( \dot{\varepsilon} \) - strain rate
\( \delta \) - relative elongation up to fracture
\( d \) - grain size
Introduction

Progress in materials science depends to a great extent on new data on the relationship between materials' structure and their properties. This conventional approach might seem rather unpromising, but the latest studies have confirmed that this old formula is still valid.

At present, much progress can be made in understanding the nature of ductility and its control. Ductility is the ability of materials to change their shape without failing under the effect of external stresses. This property is widely used in technology, in particular, in metals forming, and is the most important parameter in determining the application of metals in mechanical engineering. Materials bearing mechanical load and used in various structures must have a certain resource of ductility.

There are three main classes of materials which show marked differences in their ductility, viz., metallic materials, intermetallics, and ceramics. In metals and metal-based alloys, the relative elongation can vary from a few per cent to several dozens of a per cent, while in intermetallics it is hardly 1%, and in ceramics almost zero, and it usually fails without any signs of plastic deformation.

Investigations carried out in recent years radically changed the concept of ductility. It has been shown that the ductility of all classes of materials can be increased dozens and even hundreds of times by converting them to a superplastic (SP) state. The existing division of materials into ductile and nonductile refers only to the properties of materials under conventional test conditions and with ordinary structures. In an SP state, cast alloys, e.g., cast irons, can be even more ductile than deformable alloys, i.e., steels. Superplasticity has also been observed in intermetallics and ceramics.

Superplasticity, if understood as the ability for materials to have an abnormally large elongation, comprises a fairly wide range of phenomena, the most typical being structural superplasticity observed in microcrystalline materials and superplasticity observed during the development of phase transformations in the course of deformation, thermocycling, or under radiation. The first group is the most interesting, because, in this case, superplasticity is a universal state of materials observed at a certain microstructure, temperature and rate of deformation. This book deals only with structural superplasticity.

The main features of SP flow are (the abrupt growth of the relative elongation, which can attain hundreds or even thousands of a per cent, a remarkable decrease in the flow stress and practically no work hardening during deformation, and high strain rate sensitivity of flow stress. It is understood that these peculiarities of SP
flow are quite favourable for the process of materials forming. However, to apply this phenomenon successfully, the following problems must be solved. How can an SP state in commercial materials be obtained? How will SP treatment affect the materials performance? Is it possible to apply SP treatment in combination with other techniques to alter the physical and mechanical properties of materials?

Regarding many materials, the answers are already available; in other cases, solutions of the problems can be outlined. This book presents a detailed discussion of the factors affecting superplasticity in materials. It is shown that there is a possibility of converting alloys most widely used in mechanical engineering, including conventionally low-ductile materials, such as titanium and nickel-based superalloys as well as intermetallics and ceramics to superplasticity. Special attention is given to the effect of the structure and area of grain boundaries on the properties of materials.

The mechanism of SP flow is thoroughly analyzed and a physical model of the phenomenon explaining the variation of both the mechanical properties and structure during deformation is proposed. This model can also be used to predict new effects. Based on current knowledge of the physical nature of high-angle boundaries, a principally new postulate is offered, viz., dislocations running into grain boundaries under the temperature-rate conditions of SP flow alter the structure of grain boundaries and become nonequilibrium. As a result, grain boundary sliding which is the main mechanism of SP flow is stimulated.

The microcrystalline structure is a factor indispensable for the realization of superplasticity. The conditions for creating such a microstructure in real materials posed another important problem discussed in this book. Systematic investigations in a wide range of materials indicated that by employing the peculiarities of static and dynamic recrystallization and phase transformations, easily applicable methods of the microstructure refinement can be proposed.

Materials with micro-, submicro-, and nanocrystalline structures are of considerable interest. The book presents data on the properties of such materials and basic opportunities for "grain boundary hardening", reduction of the temperature, or increase of the rate of SP deformation.

The properties of materials after their processing in the SP state are of practical interest. The book presents systematic data on the effect of such treatment on the properties of commercial alloys. A specific microstructure is formed in materials processed under SP conditions. When intricately shaped components are manufactured by conventional forming, the strain level is different in different areas of the blank, depending on the shape. Hence, the deformation is nonuniform, which results in the microstructure heterogeneity and, consequently, in the heterogeneity of the mechanical properties of materials that is retained and in some cases even aggravated during the subsequent heat treatment. Under SP flow, the strain dependence of the material microstructure is negligible, and the material undergoes intensive "mixing" due to the development of grain boundary sliding and diffusion processes providing for a microstructure with a high structural and chemical homogeneity. The absence of cold working and texture and the retention of the microcrystalline structure enhance the ductility, impact
strength and stability of mechanical properties and decrease their anisotropy at room and lower temperatures. Thus, we can conclude that the SP flow conditions are favourable not only for the materials forming, but also for the controlled variation of their structure and physico-mechanical properties.

This book was compiled by a joint effort of several researchers and is based on results obtained by independent investigations performed in the Institute for Metals Superplasticity Problems of the USSR Academy of Sciences, Ufa. It follows the approach first described in [1.1]. However, all sections have been updated in accordance with new data reported in the world literature.

Chapters 1 and 2 were written in cooperation with R.Z. Valiev; Chapters 3, 6, and 7 with G.A. Salishchev; Chapter 4 with R.O. Kaibyshev; Chapter 5 in cooperation with M.Kh. Rabinovich; Chapter 9 with R.M. Imayev; and Chapter 10 in cooperation with N.G. Zaripov.

The author would like to acknowledge the help provided by N.V. Markushev, R.Ya. Lutfullin, R.G. Zaripova, and V.A. Valitov in the work on Chapters 5–8, respectively.

The author is also grateful to K.G. Farchutdinov and O.Sh. Gainutdinova for her assistance in preparing the manuscript for publication.
1. Structural Superplasticity

There are three conditions required for transforming a material into a superplastic (SP) state, viz., the presence of a stable microcrystalline structure \( (d < 10-15 \mu m) \), a deformation temperature exceeding \( 0.4 T_m \), and a specific range of strain rates (usually from \( 10^{-4} \) to \( 10^{-1} \) s\(^{-1} \)). In virtually all alloys the development of the SP effect has common features because of which it can be treated as a peculiar type of plastic deformation that is characterized by the specific phenomenology. Recent studies show that typical features of superplasticity can be observed not only in metals, but in intermetallic compounds and ceramics as well.

The most important difference between conventional deformation and superplastic deformation (SPD) is that in the former case, the flow stress depends on the degree of deformation and not so significantly on the strain rate. On the other hand, under the conditions of SP flow, the flow stress varies slowly as a function of the degree of deformation but depends strongly on the strain rate. Thus, structural superplasticity can be defined as the ability of metals and alloys to undergo a high degree of plastic deformation without failing under the conditions of high strain rate sensitivity of the flow stress.

1.1 Phenomenology and Influence of the Initial Microstructure

Numerous books and reviews [1.1–9] detail the typical behaviour of materials in the SP state. In this section we report the most significant results of phenomenological studies of superplastic flow which are essential for understanding the deformation mechanisms and the properties of specific industrial alloys.

One of the most distinguishing features of a specimen undergoing SP deformation is its high deformation stability, i.e., its high resistance to neck formation and grain growth during tensile deformation. According to the concepts developed by Hart (1967), the reason for such a deformation stability can be understood on the basis of the dependence of the flow stress on the strain and the strain rate. Under isothermal conditions, this relationship assumes the form

\[
\sigma = A \varepsilon^n \dot{\varepsilon}^m
\]

where \( A \) is an empirical constant; and \( n \) and \( m \) are the parameters of the material
that determine the dependence of strengthening on the degree of deformation and the strain rate.

The parameter of strain hardening of the materials whose flow stress is a slowly varying function of the strain rate (i.e., at \( m \) close to zero) can be written as

\[
   n = \frac{d \ln \sigma}{d \ln \dot{\varepsilon}}. \tag{1.2}
\]

For the materials exhibiting insignificant (weak) strain hardening, (1.1) becomes

\[
   \sigma = A\dot{\varepsilon}^m \tag{1.3}
\]

and the rate-sensitive flow stress parameter is given by

\[
   m = \frac{d \ln \sigma}{d \ln \dot{\varepsilon}}. \tag{1.4}
\]

The stability of plastic deformation is evaluated using the aforementioned parameters and by taking into account the condition of neck formation in the specimen. In the region of localization of the deformation, the strain rate and the degree of deformation are larger than those observed in the rest of the specimen. This region (i.e. neck) of the specimen is correspondingly hardened as determined by the parameters \( n \) and \( m \). Thus, a highly stable plastic deformation can be realized either by work hardening or by strengthening due to strain rate sensitivity, i.e., by ensuring high values of the coefficients \( n \) and \( m \).

In order to reveal the differences between superplastic flow and the usual plastic deformation, let us examine the mechanical properties of the alloys tested under different conditions.

It is known that plastic deformation is usually accompanied by continuous hardening during the process of tensile deformation which is followed by localized deformation, neck formation, and fracture. On the other hand, an analysis of the true tensile stress-strain curves under the conditions of SP deformation shows that soon after the initial stage, plastic deformation occurs at low and constant stresses virtually without hardening up to several hundreds and thousands of percent; sometimes, softening is observed. In this case, localization of deformation in the form of necks either ceases or occurs slowly during the entire process of tensile loading.

Furthermore, the strain rate dependence of the mechanical properties of superplastic alloys also differs significantly from that observed during usual deformation (Fig. 1.1). SP deformation is characterized by a strong strain rate dependence of the flow stress which has a sigmoidal form in the \( \ln - \ln \dot{\varepsilon} \) plot (Fig. 1.1a). Such a form of the \( \sigma - \dot{\varepsilon} \) curve makes a division into three regions possible. At low strain rates, one observes a relatively mild dependence of \( \sigma \) on \( \dot{\varepsilon} \) (Fig. 1.1a, region I) and low values of \( m \) and \( \sigma \) (Fig. 1.1b,c). With increasing \( \dot{\varepsilon} \), the strain rate dependence of the flow stress becomes stronger (more distinct), the values of \( m \) and \( \delta \) increase, and a transition to region II occurs where the effect of SP attains its maximum. The range of strain rates corresponding to region II is somewhat different for different alloys, but it is usually from \( 10^{-4} \) to \( 10^{-2} \) s\(^{-1} \).
Subsequent increase of strain rate leads to a decrease of \( m \) and \( \delta \). In region III, at high strain rates, the relative elongation \( \delta \), the flow stress \( \sigma \), and the parameter \( m \) approach the characteristic values of typical ductile materials. Such a shape of the \( \ln \sigma - \ln \varepsilon \) curve [usually referred to as a curve of superplasticity (SP)] showing three clearly distinct regions is typical for various materials existing in the superplastic state. We note that in [1.10-11], an increased strain rate sensitivity (rather than a decreased sensitivity) was observed at low strain rates where the parameter \( m \) approaches unity. Based on this, the authors proposed that the usually observed decrease of the parameter \( m \) in region I is nothing but an apparent effect due to the grain growth occurring during the process of SP deformation that is particularly rapid at low strain rates (Sect. 1.2). However, a careful study of the mechanical properties and the structure of the alloys does not support this hypothesis [1.12]. Grivas et al. [1.12] believe that in earlier studies there was an error due to the measurement of the parameter \( m \) at extremely low strains (levels of deformation) that are not characteristic of the stage of stable SP flow. Confirming the \( \ln \sigma - \ln \varepsilon \) relationship at low strain rates is an important aspect requiring additional studies.

The correlation between the strain rate dependences of \( \delta \) and \( m \) is an important experimentally established fact. The results obtained when testing numerous alloys show that the transition to SP occurs at \( m \) exceeding 0.3 and that lower values of \( m \) retain the usual deformation during which \( \sigma \) and \( \delta \) are slowly varying functions of the strain rate.

We note that Hart's mechanical model of visco-elastic flow offers only a qualitative explanation for the behaviour of superplastic materials, and the attempts
made to establish the quantitative relationship between the coefficient $m$ and ductility have given an ambiguous result [1.1-4]. Nevertheless, the qualitative relationship between $\delta$ and the coefficient $m$ that forms the most important characteristic of superplastic materials has been established for almost all important metals and alloys in their superplastic state.

There have been numerous studies concerning the methods for determining the coefficient $m$ [1.1-5, 13]. It is determined from the slope of $\ln \sigma - \ln \dot{\varepsilon}$ curve or according to the method of abruptly changing the strain rates during the process of deformation using different methods of calculations and extrapolation of the loading power versus deformation curves using the test data concerning stress relaxation. This problem has received a lot of attention because of the difficulties in determining the "true" value of $m$. Frequently, the values of $m$ determined by different methods are qualitatively correlative with relative elongation, but, quantitatively, significant differences are observed.

The differences observed in the absolute value of the coefficient $m$ measured according to different methods are due to a number of factors which include the initial microstructural state of the material, the structural changes occurring during the process of tensile deformation, and the level of deformation (strain) at which $m$ is determined. Our results [1.13] show that the shape of the stress-strain curves has great significance under the conditions of SP flow. The point is that the measured value of $m$ is significantly affected by the magnitude and the sign of the coefficient of the strain hardening $n$ which depends on the shape of the true deformation curves. The experiments show that, when determining the coefficient $m$, it is necessary to analyze the true curves of tensile deformation at different strain rates and carry out measurements only at the points where the coefficient $n$ has the same sign for both the comparable strain rates. This situation requires a detailed study of the true curves of tensile deformation at different strain rates. However, if one opts to carry out the entire set of experiments, the very essence of determining the coefficient $m$ as a parameter simplifying the evaluation of the superplastic behaviour of materials is lost from a practical standpoint.

There is another problem in measuring the "true" value of the coefficient $m$, namely, the nonuniformity of SP deformation. In many studies the formation of one or several necks and their "wandering" along superplastically deformed samples were observed. The localization of deformation at the level of individual grains is also typical of SP flow. The studies performed on samples with networks marked on their surfaces give evidence of a significant nonuniformity of deformation of a few dozens of microns when the strains of the neighbouring areas can differ by 50--60% from the average strain of the specimen. The local values of strain rate and coefficient of strain rate -- stress sensitivity also vary in this case. The measured values of $m$ are average; hence, they depend on the distribution of local deformations.

The facts show that at the present time no universal method can be selected for determining the absolute value of the coefficient of strain rate sensitivity of the flow stress, and the measured coefficient $m$ must be treated not as a material constant, but as a structure-sensitive parameter whose specific value depends not
only on the procedure followed for its determination, but also on the strain of the specimen and the strain rate. Thus, SP flow is characterized by the following features.

1) Large values of relative elongation of the specimens subjected to tensile loading and a significantly stable plastic deformation process owing to the increased strain rate sensitivity of the flow stress;
2) a relatively low flow stress and the absence of noticeable strain hardening during the process of deformation;
3) a considerable strain rate dependence of the flow stress, the elongation, and the parameter \( m \).

The parameters of superplastic flow \( \sigma, \delta, \) and \( m \) and the location of the optimum range of strain rates are determined by the structure of the alloy and the temperature of deformation. An analysis of the effect of various factors on SP flow is given below.

**Temperature**

SP deformation is characterized by a significant dependence of the mechanical properties on the deformation temperature. Increasing the temperature leads to a decreased flow stress and to a displacement of the optimum strain rate range of SP deformation (Fig. 1.1, region II) towards higher values of \( \dot{\varepsilon} \). However, such an effect of temperature is observed only in the range corresponding to a stable microcrystalline structure. If a significant grain growth occurs with increasing temperature or the stability of the microstructure is lost as a result of the transformation of the material into a single phase condition, the SP effect decreases abruptly or disappears completely.

The temperature dependence of the rate of superplastic deformation can be described by Arrhenius' equation

\[
\dot{\varepsilon} = c \exp\left(-\frac{Q}{RT}\right),
\]

where \( c \) is a constant; \( Q \) is the activation energy; and \( R \) is the universal gas constant.

A similar relationship can be used in describing the effect of temperature on the flow stress

\[
\sigma = k \exp\left(\frac{Q}{RT}\right),
\]

where \( k \) is a constant.

The activation energy is an important parameter of SP flow that reflects the nature of the thermally activated processes controlling the deformation rate. The magnitude of \( Q \) is usually determined by tests at a constant stress or at a constant strain rate within a certain temperature range of deformation. The activation energy is experimentally determined from the slope of the \( \ln \dot{\varepsilon} = 1/T \) or \( \ln \sigma - 1/T \) plots, depending on the test conditions. However, the preexponential terms in (1.5, 6) usually depend on the temperature and, consequently, the so-called
Table 1.1. Values of the activation energy for SP flow at three ranges of strain rates and activation energy for volume $Q_{vol}$ and grain boundary $Q_{gb}$ diffusion in kJ/mole for some alloys. Data for the self diffusion of the indicated component of the alloy is given. From [1.1]

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Activation energy for SP flow in the regions</th>
<th>$Q_{vol}$</th>
<th>$Q_{gb}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>I</td>
<td>II</td>
</tr>
<tr>
<td>Zn-0.4%Al</td>
<td>59</td>
<td>42</td>
<td>84</td>
</tr>
<tr>
<td>Zn-22%Al</td>
<td>74</td>
<td>79</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>96</td>
<td>70</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>119</td>
<td>78</td>
<td>–</td>
</tr>
<tr>
<td>Sn-38%Pb</td>
<td>80</td>
<td>50</td>
<td>80</td>
</tr>
<tr>
<td></td>
<td>84</td>
<td>53</td>
<td>–</td>
</tr>
<tr>
<td>Sn-35%Bi</td>
<td>–</td>
<td>46</td>
<td>88</td>
</tr>
<tr>
<td>Cd-5%Pb</td>
<td>59</td>
<td>42</td>
<td>–</td>
</tr>
</tbody>
</table>

apparent activation energy which somewhat differs from the true magnitude of $Q$. In spite of this conditional nature, the measurements of the activation energy are of great importance in analyzing the nature of SP flow, and some respective data can be found in [1.1–9] (Table 1.1). In region II the magnitude of $Q$ is usually close to the activation energy for grain boundary diffusion, but increases with an increasing or decreasing strain rate; in region III it usually approaches the activation energy for volume diffusion.

Grain Size

Among the structural factors affecting the SP effect, grain size is the most important since a stable microcrystalline structure ($d < 10–15\mu m$) is an essential condition for realizing structural superplasticity.

Since in the near-single phase alloys it is possible to produce equiaxial grains having a size ranging from a few microns up to several hundreds of microns, one can follow the transition from the usual deformation to the superplastic flow with increasing microstructural refinement. For example, in the case of the Zn-0.4%Al alloy, it was shown in [1.3] that at $d > 10\mu m$, the dependence of $\sigma_{0.2}$ on $d$ (Fig. 1.2) obeys the Hall-Petch relationship according to which $\sigma \sim d^{-1/2}$. However, at $d < 10\mu m$, the grain size dependence of the yield stress follows a reverse trend and $\sigma_{0.2}$ decreases with decreasing $d$.

With increasing refinement of the structure, the ductility of the Zn-0.4%Al alloy and the strain rate dependence of its properties undergo changes. In particular, abrupt changes in the relative elongation are observed as the grain size changes from 10 to 1\mu m; increases from 25 up to 400% (Fig. 1.2) and, simultaneously, the characteristic strain rate dependence of the properties of the alloy becomes distinct. The results of numerous studies of the relationship between the flow stress and the grain size are summarized in [1.1–4]. It is shown that during superplastic deformation, the following form exists:
Fig. 1.2. Dependence of the yield stress of the Zn-0.4%Al alloy on the grain size $d$ at 20°C and $\dot{\varepsilon} = 10^{-3}$ s$^{-1}$ [1.3]

\[ \sigma \sim d^a, \quad (1.7) \]

where $a = 0.7 - 2$ and, most frequently, $a = 1$.

It was established that at a constant $\sigma$ the effect of grain size on the strain rate in the second region can be expressed as

\[ \dot{\varepsilon} \sim d^{-b}, \quad (1.8) \]

where $b = 2 - 3$, most frequently with $b$ being closer to 2.

A change in the grain size has a significant effect also on the strain rate dependence of the flow stress, i.e., decreasing the grain size makes the optimum $\dot{\varepsilon}$ II range, within which the SP effect is developed, shift towards higher strain rates. The maxima of the $m - \dot{\varepsilon}$ and $\delta - \dot{\varepsilon}$ plots shift towards higher $\dot{\varepsilon}$; moreover, the maximum value of $\delta$ increases.

The role of grain size refinement is clearly displayed in SP alloys with superfine grains. In recent years a number of ways of producing submicron-grained structures with a grain size of about 0.1 μm has been developed (Chap. 3), and data on the SP deformation of these alloys have been published in [1.14-17]. Va­liev et al. [1.14] were the first to report an abrupt drop of a temperature at which SP flow occurs. In the Al-4%Cu-0.5%Zr alloy, a decrease in SP temperature from 500 to 220°C was observed when grain size reduced from 8 to 0.3 μm at a constant strain rate of $3 \times 10^{-4}$ s$^{-1}$. The values of SP flow parameters remained almost unchanged – the coefficient $m$ was close to 0.5, flow stress was about 20–30 MPa and relative elongation to fracture was more than 400%. The said drop in the SP temperature can also be observed in submicron-grained Ti-based and Mg-based alloys and steels (Chaps. 4–6).

However, the submicron-grained structure is unstable, and intensive grain growth occurs at temperatures higher than 0.4 $T_m$. Therefore, the studies [1.16, 17] of mechanically alloyed specimens in which a structure with a grain size of 0.2 μm can be retained up to 0.7–0.8 $T_m$ due to disperse oxide particles are of great interest. It was established that typical SP behaviour is observed at 500°C in a strain rate range from $10^6$ to $10^1$ s$^{-1}$ (these strain rates are close to blow). It is evident that SP deformation of submicron-grained alloys is of great importance for practical application because of the possibility of abruptly increasing
the production output and significantly prolonging the service life of the punch
tackle.

In addition to the study of the SP deformation of submicron-grained alloys,
the investigations of the SP of coarse-grained materials are also interesting. Typ¬
ical features of superplasticity (SP) in coarse-grained alloys were observed in
[1.1, 18, 19]. There are apparently several reasons underlying such unusual be¬
haviours of these alloys. In particular, during the deformation process of coarse­
grained titanium alloys a substructure forms within the grains whose behaviour
is probably similar to that of the fine-grained structure of SP materials (Chap. 6).

Furthermore, in specimens of the Al-Tl and Al-Ge alloys, superplasticity is
observed at $d = 100 - 200 \mu m$ [1.19]. In this case, no substructure was observed
during the deformation process. At the same time, a correlation exits between
the initial porosity of the alloys (caused by phase transformations during the heat
increase to test temperature) and the relative elongation under the conditions of
superplastic flow. The maximum ductility was obtained in the Al-0.4%Ge alloy
in which the initial porosity attained the highest level (approximately 0.8 vol.%).
Kuznetsova et al. [1.19] showed that the SP effect in the Al-Ge alloys is due
to the presence of porosity and facilitates the development of the combination
of mechanisms that operate in the common superplastic alloys; and because the
porosity is maintained at a constant level, it does not lead to a fracture of the
material.

**Grain Size Distribution**

When analyzing the effect of the structural characteristics on the superplastic
behaviour, one usually refers to an average grain size. However, in individual
cases the size of the crystals in the experimental alloy can vary over an order
of magnitude or more. This is particularly true for the industrial alloys where,
during the process of obtaining fine grains by severe prior plastic deformation
(working), coarse-grained zones are developed within the fine-grained structure.
Naturally, the presence of different grain sizes changes the properties of the
alloys under the conditions of SP flow, and the development of coarser grains
expected to affect the SP effect adversely.

The behaviour of the industrial 1420 aluminium alloy (Al-5%Mg-1.8%Li-
0.12%Zr) clearly illustrates this fact. The alloy was obtained in three conditions:
with an equiaxial fine-grained microstructure ($d = 6 \mu m$), with a coarse-grained
microstructure ($d = 75 \mu m$), and with a mixed structure in which equiaxial fine
grains ($d = 6 \mu m$) are present along with coarse elongated grains. In this condition,
the ratio of the areas of the fractions of the fine and the coarse grains was found
to be 1:1.

The dependence of $\delta, \sigma$, and $m$ on the microstructure during the deformation
of the 1420 alloy is shown by the data below ($t_{def} = 450^\circ C$ and $\dot{\varepsilon} = 10^{-3} s^{-1}$):
These data show that the features of the SP state appear even in the presence of a mixed structure in the alloy. However, in this case, there is a certain increase in the flow stress and a decrease in the relative elongation. At the same time, the range of strain rates corresponding to that of SP shifts towards the region of lower strain rates.

The studies carried out on the Zn-22%Al and Ti-6%Al-4%V alloys and brasses [1.13, 20] showed that the location of the optimum range of strain rates and the value of $m$ depend on the nature of grain size distribution. In [1.21] approaches that make it possible to predict the properties of the alloy are discussed. These take into account the volume fractions of the grains of different sizes, under the assumption that their contribution to the flow stress of SP is additive. A comparative study of the results calculated according to this approach shows a satisfactory agreement with the experimental values of the coefficient $m$, but there is a considerable difference in the values of the flow stress. The latter is probably caused by the interaction of deformation processes in the areas with coarse and fine grains.

The presence of microheterogeneities in the form of elongated particles along the prior boundaries of the grains of a high-temperature phase can be considered to be one of the types of bimodal structures arising during the process of deformation. In Zn-22%Al alloy and brasses, the presence of a network of microinhomogeneities in the boundaries of the coarse grains with the background of an ultrafine-grained structure significantly decreases the relative elongation and increases the flow stress under the conditions of SP [1.1, 20, 22]. Destruction of the network of these boundaries by prior plastic deformation leads to a decrease of $\sigma$ and in increased $\delta$.

**Phase Composition**

Grain size is an important, but not the only structural characteristic determining the properties of superplastic alloys. Chemical and phase compositions have a significant effect on their structure and properties under the conditions of superplastic flow. The relationship between the chemical composition and the SP effect will be examined in greater detail when the effect of alloying on the properties of concrete industrial alloys are analyzed. It is known [1.2, 3] that chemical composition has an indirect effect on SP through the microstructure, i.e., by creating the conditions required for obtaining a stable fine-grained structure. The effect of phase composition on the phenomenon of SP flow has also been examined primarily from the standpoint of microstructural stability [1.1–9]. In fact, this is the most evident aspect of the effect of phase composition on SP. It is not accidental that the very first observations and studies on SP were made on alloys having

<table>
<thead>
<tr>
<th>$d$ [μm]</th>
<th>6</th>
<th>75</th>
<th>Mixed</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\delta$ [%]</td>
<td>720</td>
<td>210</td>
<td>390</td>
</tr>
<tr>
<td>$\sigma$ [MPa]</td>
<td>5</td>
<td>20</td>
<td>13</td>
</tr>
<tr>
<td>$m$</td>
<td>0.55</td>
<td>0.22</td>
<td>0.40</td>
</tr>
</tbody>
</table>
nearly an identical proportion of the phases of the eutectic or the eutectoid composition. In such materials, a fine-grained structure is most easily obtained and retained. It is readily apparent that the structural stability of the alloys increases with increasing content of the second phase; therefore, it is easier to convert multiphase alloys into the SP state as compared to single-phase alloys, and it is virtually impossible to attain the SP state in ultra-pure metals using the principles of structural superplasticity. However, microstructural stability and the grain size of alloys are not the only factors reflecting the effect of phase composition on SP; the deformation characteristics of the phases have a significant effect on the superplastic behaviour.

Let us now examine this aspect in detail. The deformation characteristics of the phases depend on their chemical composition, the type of their lattice and the homologous temperature of deformation. It might appear that knowing these characteristics, one can predict whether a multiphase alloy can be deformed superplastically. However, the actual behaviour of such alloys is much more complex, because interaction of the phases occurs during the deformation process, which must be taken into account. For instance, during the process of deformation, grain growth of the phases cannot occur without interdiffusion of these components, consequently, the diffusion characteristics of the phases can have a significant effect on SP. Furthermore, a change in the phase composition leads to a change in the structure and in the extent and the fraction of the inter-phase boundaries in the alloy. The fact that this is significant for superplastic deformation follows from the differences in sliding at the grain boundaries and the interphase boundaries [1.1]. Finally, the predominant slip systems in the phases depend on the phase composition. It was established that in the ultrafine-grained Zn-Al alloys, the active slip systems of the zinc-based beta-phase depend on the content of the alpha-phase [1.23]. Increasing the content of the alpha-phase activates the nonbasal slip systems in the beta-phase. This can be attributed to the fact that it becomes easier to generate the \((c+a)\) type dislocations at the interphase boundaries than at the grain boundaries in the beta-phase.

Detailed and systematic studies on all these aspects have not yet been carried out. However, it is already clear that, from the standpoint of phase composition, the optimum conditions for the development of SP do not simply imply obtaining a 1:1 ratio of the phases. Depending on deformation characteristics of the phases, their optimal ratio (with respect to superplastic deformation) can increase or decrease. A special investigation was made on this aspect using alloys of the Zn-Al system containing 0.4–72 wt.%Al [1.3, 22]. The homologous temperatures for the alpha and beta phases are significantly different in these alloys and, consequently, at 250°C, the possibility of plastic deformation is considerably greater in the beta-phase than in the alpha-phase. Thus, as the strain rate increases, the Zn-10%Al alloy exhibits a greater relative elongation than the alloy of the eutectoid composition. The importance of the deformation characteristics of the phases becomes particularly evident at low temperature deformation where stability of grain size is less important. In this case, the zinc-rich alloys are most ductile in spite of the fact that, from the standpoint of superplastic deformation, their
structure is less favourable than that of the eutectoid alloy. The behaviour of the alpha-phase in the alloys containing more than 50 vol.% beta-phase is somewhat similar to that of the hard particles present in a ductile matrix.

Similar results were subsequently obtained during a study of the effect of the phase composition of alloys of the Cu-Zn system on their SP behaviour [1.24]. Thus, the role of the phase composition of the alloys apparently does not only reside in evolving the optimum microcrystalline microstructure having a highly stable grain size at $T > 0.4 T_m$ in the formation of two-phase microstructures, it is also necessary to take into account the deformation characteristics of each phase and the interaction between the phases during the deformation process.

**Texture**

The effect of texture on the properties of alloys is traditionally related to the development of preferred grain orientation in the polycrystalline matrix. The mechanical properties of single crystal depend on the orientation of the lattice with respect to the direction of load application due to which, in the presence of preferred grain orientation, polycrystalline materials exhibit anisotropy of properties.

Under the conditions of superplastic deformation, this effect was studied for the first time on the Zn-0.4%Al alloy [1.25]. It was established that $\sigma$, $\delta$, and the coefficient $m$ vary considerably depending on the direction of cutting the specimens relative to the rolling direction. According to Naziri and Pearce [1.25], this effect is associated with the presence of a crystallographic texture in the Zn-0.4%Al alloy. Subsequently, anisotropy of properties under the conditions of SP was observed in a number of alloys, viz., Zn-Al [1.26, 27], aluminium bronze [1.28], brasses [1.20], Sn-Bi [1.29], etc. However, the results cannot always be considered as a consequence of the presence of preferred grain orientation. The microstructure of alloys (in particular, industrial alloys) in which superplasticity is observed shows a certain degree of structural nonuniformity (elongation of grains and directionality in the distribution of precipitates and phases) because of certain aspects of the sample preparation and the presence of impurities and inclusions; such a nonuniformity can influence the SP effect. Many investigators consider that anisotropy of properties is a result of anisotropic distribution of the particles of a second-phase in the structure. For example, it was established [1.20] that microstructural anisotropy as well as the crystallographic texture of the L59 brass cause nonuniformity of the properties in relation to the rolling direction. Apparently there is a cumulative effect of these factors on the anisotropy of properties even in other cases, but this aspect has not yet been studied in sufficient detail.

It should be noted that anisotropy of relative elongation alone is usually observed and the flow stress is isotropic [1.27]. Anisotropy of the flow stress was absent in spite of the presence of a texture, and its dependence on the direction in which specimen cutting was carried out was seen only in the case of an alloy having grains elongated in the rolling direction. The absence of anisotropy of
the flow stress makes it somewhat difficult to follow the effect of textures on the properties of the alloys; therefore, some investigators believe that preferred grain orientation does not change the SP effect. However, the absence of anisotropy of the flow stress can be one of the specific features of the effect of texture on SP and this, by no means, implies the absence of such an effect. In order to study the "pure" effect of preferred grain orientation on the superplasticity of an alloy, it is necessary to carry out property measurements in a textureless condition and in the presence of different textures maintaining identical microstructures of the alloy. Such studies have been carried out on the Zn-22%Al and VT6 titanium alloys [1,3,29,30].

The most detailed study on the effect of texture was carried out on the Zn-22%Al alloy in which, using the specific features of the monotectoid decomposition during quenching and controlling the regimes of rolling, it is possible to obtain a wide variety of conditions ranging from the textureless state up to a sharp preferred orientation within the beta-phase alone as well as in both alpha- and beta-phases. Table 1.2 shows the processing conditions selected for the experimental study. All the processing regimes yielded equiaxial structures, and an equal grain size (approximately 0.5 µm) was obtained by varying the duration of annealing at 250°C.

A comparative study of the properties of the textures and the textureless alloy in the as-quenched state makes it possible to reveal the effect of texture on the mechanical properties in the "pure form", because as a final treatment, quenching makes preferred grain orientation the main structural parameter distinguishing between state 1 and 2 (Table 1.2). As seen from Table 1.2, the presence of a texture leads to a decrease in the flow stress over the entire experimental range of temperature and strain rate.

An analysis of the true stress-strain curves shows that the difference in their magnitudes retained up to \( \varepsilon = 150\% \) and more. The relative elongation of the quenched textured alloy is significantly greater than that of a texture-free alloy (Fig. 1.3).

![Graphs](image.png)

**Fig. 1.3.** Dependence of relative elongation (a) and the coefficient \( m \) (b) on Zn-22%Al at 250°C on the strain rate [1,1]. The curves 1–4 correspond to the states of the alloy indicated in Table 1.2.
Table 1.2. Effect of the texture on the flow stress $\sigma_{40}$ at $\dot{\varepsilon} = s^{-1}$ of the Zn-22% Al alloy. From [1.27]

<table>
<thead>
<tr>
<th>Number of the state of the alloy</th>
<th>Mode of producing structure</th>
<th>Type of texture</th>
<th>$\sigma_{40}$ [MPa]</th>
<th>$2.8 \times 10^{-4} s^{-1}$</th>
<th>$2.8 \times 10^{-2} s^{-1}$</th>
<th>$2.8 \times 10^{-1} s^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\alpha$-phase</td>
<td>$\beta$-phase</td>
<td>20°C</td>
<td>150°C</td>
<td>250°C</td>
</tr>
<tr>
<td>1</td>
<td>Quenching, rolling at 250°C, annealing at 350°C for 5 h, and quenching</td>
<td>none</td>
<td>none</td>
<td>183</td>
<td>21</td>
<td>2.5</td>
</tr>
<tr>
<td>2</td>
<td>Quenching, rolling at 20°C, annealing at 350°C for 5 h and quenching</td>
<td>(001) [100] + (110) [112]</td>
<td>(1010) [0001] + (1210) [1010]</td>
<td>114.5</td>
<td>15.3</td>
<td>2.2</td>
</tr>
<tr>
<td>3</td>
<td>Quenching and rolling at 20°C</td>
<td>(001)[100]+ (120)[212]</td>
<td>(1010) [0001]+ (0001)+ +24° TD+RD[1010]</td>
<td>88.6</td>
<td>3.2</td>
<td>1.04</td>
</tr>
<tr>
<td>4</td>
<td>Quenching and rolling at 250°C</td>
<td>none</td>
<td>(0001)</td>
<td>101.0</td>
<td>9.9</td>
<td>1.26</td>
</tr>
</tbody>
</table>
Texture influences the location of the optimum range of strain rates for realizing the SP effect. It can be seen from Fig. 1.3 that the maxima of the parameters $m$ and $\delta$ of the alloy existing in state 2 shift towards higher strain rates as compared to those of the alloy corresponding to state 1. Thus, at a given microstructure, the presence of a texture in the Zn-22%Al alloy leads to a decreased flow stress, an increased relative elongation, and a shift of the optimum strain rate interval towards higher strain rates. With a decreasing test temperature, the effects of textures are not only retained, but are intensified (Table 1.2). Thus, after quenching, at 205°C and $\dot{\varepsilon} = 2.8 \times 10^{-4} \, s^{-1}$, the value of $\delta$ of the textured alloy is 1.3 times higher and $\sigma$ is 1.25 times less than those of the textureless alloy. At 150°C, these values increase to 2.1 and 1.5 times, respectively. The optimum strain rates corresponding to states 1 and 2 differ by 2–3 times at 250°C and by 15 times at 150°C. The difference in the room temperature properties of the alloy in states 1 and 2 is still greater, but its quantitative evaluation is difficult because the optimum range of strain rates corresponds to $10^{-4} \, s^{-1}$.

As a result of prestraining of a quenched texture-free alloy, the SP effect in it increases significantly [1.1, 3]. Portnoi et al. [1.20] attribute such a change in the properties to the destruction of the network of the elongated particles along the grain boundaries of the high temperature phase and not to the development of textures during the course of deformation. Our studies [1.27] show that structural inhomogeneity affects the flow stress and the relative elongation during the initial states of deformation but has virtually no effect on the strain rate dependence of these mechanical properties. The results in Table 1.2 and Fig. 1.3 show that in the rolled alloys (states 3 and 4), the relative elongation increases and the flow stress decreases significantly as compared to the properties obtained in the as-quenched alloys. Even in this case one observes a shift of the strain rate range corresponding to the SP effect in the alloy having preferred grain orientation, i.e., the observed change in the properties of the alloy after rolling is not only due to the destruction of the microstructural heterogeneities, but is also due to the texture development during the process of rolling.

The presence of a texture causes a significant difference between the properties of the alloy existing in states 3 and 4 that are distinguished only by the nature of the rolling textures. The flow stress of the cold rolled alloy and the relative elongation of the former is higher. In this case, the optimum strain rate range of the alloy existing in state 3 is shifted towards the region of higher strain rates. As in the case of alloys in the as-quenched state, the effect of texture on the properties of the rolled alloys increases significantly with a decreasing test temperature.

The universal nature of the effects of preferred grain orientation on the properties of the alloys under the conditions of superplastic flow is also confirmed by the studies carried out on the titanium based VT6 alloy [1.29, 30], which was obtained in two states, viz., without any texture and with a strong texture, using different combinations of the regimes of compressing and rolling at 800–900°C. The microstructure was equiaxial; an identical grain size (approximately 4 μm) was obtained in both states by varying the duration of annealing. Thus, the basic
requirement of identical microstructures and different textures could be met in these two states.

Tensile tests showed that the flow stress of the textured alloy is less than that of the textureless alloy. Such an effect of texture was noticeable at the test temperatures ranging from 700 to 900°C, because the difference in the flow stress increases with decreasing temperature of deformation. The plasticity of the alloy also shows a significant dependence on the texture. The relative elongation of the textured alloy is greater than that of the texture-free alloy and these differences also increase with decreasing test temperature. Thus, at 900°C the relative elongation of the textured alloy is only 1.06 times higher than that of the textureless alloy, whereas at 700°C the plasticity of the textured alloy is three times higher. A study of the strain rate dependence of $m$ showed that the optimum strain rates of the textured alloy were displaced towards the region of higher strain rates and that the extent of this displacement increased with decreasing test temperature.

The established effects of preferred orientation on the superplastic flow are most unusual and were not reported earlier. However, there are some published experimental data which can also be attributed to the effect of texture on the properties. For example, the studies of Benedek and Doherty [1.31] showed that with an increasing degree of prestraining, the grain size increases instead of decreasing. In this case, the relative elongation increased and the flow stress decreased. Similar results concerning the effect of prestraining on the properties of brasses under the conditions of SP were reported in [1.5].

The observed variations of the characteristics of SP flow can be attributed to the changes occurring in the structure of grain boundaries of a polycrystalline material in the presence of crystallographic textures. There undoubtedly exists a relationship between the texture and structure of grain boundaries, because, based on the orientation distribution function obtained from the texture data, it is possible to evaluate the distribution function of the boundaries with respect to the characteristic geometric parameters (in particular, with respect to the angles of misorientation between the grains). The structure of grain boundaries depends on its geometric parameters. The grain boundary processes playing a specific role in the phenomenon of superplasticity will be examined in Sect. 2.2.2.

The State of Grain Boundaries

In the structure of ultrafine-grained superplastic materials, the total grain boundary area is extremely high; the grain size essentially characterizes the reciprocal of the grain boundary area per unit volume. In this case, grain boundaries act not only as the geometric surfaces separating grains of different orientations, but also as important structural elements determining the properties of the polycrystalline material under consideration. Unfortunately, this aspect has not been analyzed in detail in studies on the phenomenon of structural superplasticity, mainly because of the nonavailability of adequate data on the behaviour of grain boundaries during plastic deformation processes. However, the progress made
In recent years in understanding the structure and properties of high-angle grain boundaries (Sect. 2.2) allows a new direction of experimental design and analysis in revealing the role of the state of grain boundaries in the development of SP deformation.

In this context, the results of investigations carried out on the superplastic behaviour of the magnesium alloy MA8 containing twins are of interest [1.32]. After being rolled and annealed, this alloy exhibits superplasticity at 400°C and $d \approx 10\mu m$. Twins were introduced into the structure of the alloy by cold working. After treatment, more than 75% of the grains showed deformation twins and their number remained unaltered during the process of heating up to the temperature of SP flow. The presence of twinned regions can be considered as an additional refinement of the structure; taking the twin boundaries into account, the grain size of the alloy is approximately $3\mu m$. As mentioned above, a decrease in the grain size often makes it easy to realize the SP effect and facilitates a shift of the range of strain rates corresponding to its development towards higher strain rates. However, as shown by results of mechanical testing, a different picture emerges in the alloy containing twins, i.e., the flow stress increases and the maximum values of the parameters of plasticity, viz., $\delta$ and the coefficient $m$ are displaced towards lower strain rates. Structural studies make it possible to delineate the reasons underlying the observed variation of mechanical properties. Sliding does not occur along the twin boundaries and, at the same time, twins facilitate accumulation of dislocations in the structure. Twin boundaries are high-angle boundaries but belong to a special type of grain boundaries (Sect. 2.2.2) exhibiting peculiar properties, i.e., they do not act as dislocation sinks. Thus, the results directly indicate the important role of the structure of boundaries in the development of superplasticity.

In addition to the structure of grain boundaries, the type and concentration of impurities and segregation have a decisive effect on the grain boundary processes. Thus, it can be expected that they have a significant effect on the SP flow also. This hypothesis is supported by a number of recent experimental data.

In the sintered aluminium alloy SAS-I-50 (Al-27.1%Si-5.5%Ni-0.46%Fe-1.26%Al2O3), it is easy to obtain a microstructure having a grain size of 1.5-4 $\mu m$. This microstructure is stable up to 550°C ($0.95 T_m$). However, superplastic flow is not observed. Its deformation is characterized by $\sigma > 50$ MPa, $\delta < 30\%$, and $m < 0.3$. We emphasize that the alloys having a composition close to that of the aforementioned alloy and an analogous microstructure exhibit all the features of SP under identical conditions of testing. Probably, the absence of SP in the SAS alloy is caused by the obstructed movement of the boundaries that is caused either by the oxide film formed along the grain boundaries during the preparation of the alloy or by the Al2O3 particles. As a consequence, the operation of the characteristic deformation mechanisms of superplastic flow becomes impossible.

Myshlyaev et al. as indicated in [1.1] observed the disappearance of the SP effect in the Zn-0.4%Al alloy after aging. It was established that aging the alloy for a period of 6 months leads to a state of low plasticity ($\delta = 30\%$ and $m < 0.2$), although it has virtually no effect on the grain size (approximately $1 \mu m$). It can
be concluded that a change in the impurity concentration occurs at the grain boundaries. As a result, the grain boundaries can no longer act as effective dislocation sinks and, consequently, the progress of grain boundary sliding (GBS) becomes extremely difficult. In this context, the results on the model aluminium alloys Al-2 at.%Cu-0.16 at.%Zr and Al-2 at.%Mg-0.16 at.%Zr [1.33] are of interest. There are quasi-single phase alloys. Copper and magnesium enter the solid solution and zirconium is present mainly in the intermetallic compound Al₃Zr. A microstructure having a grain size of 7 μm and a uniform distribution of the particles of the second phase Al₃Zr was obtained using a special treatment.

The data in Table 1.3 show that for a given microstructural state and under identical test conditions the flow stress of the copper containing alloy is significantly less than that of the magnesium containing alloy, but the relative elongation is considerably higher.

Table 1.3. Structural data and superplasticity parameters of aluminium alloys Al-2 at.% Cu-0.16 at.%Zr (1) and Al-2 at.%Mg -0.16 at.%Zr (2) at a strain rate of \(1.7 \times 10^{-3} \text{ s}^{-1}\) \((d_0 = 7 \mu \text{m})\). The numerator and the denominator indicate the data obtained at \(T_{\text{def}} = 773 \text{ K}\) and \(T_{\text{def}} = 723 \text{ K}\), respectively.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>(l [\mu \text{m}]^a)</th>
<th>(d_{50} [\mu \text{m}])</th>
<th>(m)</th>
<th>(\delta_{\text{max}} [%])</th>
<th>(\sigma_{50} [\text{MPa}])</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.25</td>
<td>8/7</td>
<td>0.53/0.40</td>
<td>900/400</td>
<td>4/9</td>
</tr>
<tr>
<td>2</td>
<td>0.23</td>
<td>9/7.5</td>
<td>0.33/0.27</td>
<td>360/190</td>
<td>10/21</td>
</tr>
</tbody>
</table>

*aRepresents the interparticle spacing.

An optical microscopic study of the structure arising during the process of deformation did not reveal significant differences between the alloys. The obtained differences in the mechanical properties could be explained only by conducting electron microscopy of the structure of the grain boundaries. It was established that the boundaries present in the copper-containing alloy possess a greater capacity to absorb the lattice dislocations than those existing in the magnesium-containing alloy (Sect. 2.2).

*Karpov* et al. [1.34] studied the SP effect in berillium-yttrium alloys. Due to low solubility of yttrium in beryllium, its addition leads to the precipitation of the YBe₁₃ compound along the grain boundaries which, in turn, stabilizes the fine-grained structure. According to existing concepts, this stabilization must increase the SP effect. However, in spite of the increased micro-structural stability, superplasticity of the berillium-yttrium alloys is not intensified; in fact, upon increasing the yttrium content beyond 0.15%, the magnitude of \(m\) abruptly decreases to 0.1–0.2 and the relative elongation decreases correspondingly. Such an effect of alloying is related to the changes occurring in the properties of grain boundaries due to precipitation [1.34].

The data indicate the importance of the state of the grain boundaries in the development of superplastic flow and show that the presence of an ultrafine-grained structure is a necessary, but not a sufficient condition for inducing the SP
effect. Furthermore, it is important to consider the specific features of the structure and the state of the grain boundaries and their behaviour (sliding, migration) in the microstructure of the superplastic materials which facilitate the development of the processes determining SP deformation. However, it is necessary to conduct further studies for establishing such parameters of the structure and the state of the grain boundaries that would ensure the best indices of the effect of structural superplasticity.

1.2 Structure Evolution and Fracture Modes

A study of the structural changes occurring during SP flow is important for two reasons. On the one hand, it ensures evaluation of the specific features and nature of the process of SP flow, and, on the other, it permits one to predict the properties obtainable after SP deformation. Up to now, there have been numerous studies on the microstructure, the dislocation structure, the texture evolution, the deformation relief, and the porosity appearing in various metals and alloys in the superplastic state [1.1–9]. Let us examine the basic results in order to evaluate the present state of the problem.

Microstructure

Retention of the equiaxial grain shape after SP deformation is one of the most important features established thus far. Even after attaining several hundred percent elongation, the specimens deformed in the region of the maximum strain rate sensitivity of the flow stress retain to a large extent an equiaxial grain structure. This result has been confirmed on virtually all superplastic materials. In addition, a metallographic texture (grain elongation/fibrous structure) initially present in an alloy usually decreases during deformation. There are two points of view on the observed retention of equiaxial grain shape. According to the first theory, elongation of the grains occurs during deformation, but it is eliminated by the migration of grain boundaries [1.2, 3, 35]. According to the second theory, the grains remain equiaxial due to their unusual movement during which they are redistributed as a whole with a replacement of their neighbours (Sect. 2.1.1). Evidently, both these processes can occur during superplastic deformation.

Grains remain equiaxial not only when carrying out deformation in region II, but also when the strain rate is changed. However, a significant increase in the strain rate usually leads to elongation of grains and sometimes to grain refinement because of the loss of superplasticity in the material. Lee [1.35] and Valiev and Kaibyshev [1.36] observed the development of grain elongation even when deforming at low strain rates. This effect is most pronounced in alloys having an hcp structure, and its nature is controlled, evidently, by the specific features of the mechanisms operating in region I [1.37].