PLASTICITY AND ELASTICITY
OF CRYOCRYSTALS

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HANDBOOK

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PREFACE

In this handbook, the data on mechanical behavior and elasticity are collected, peculiar to the simplest forms of the following molecular crystals:
- inert elements – argon, krypton, xenon, and neon
- molecular substances formed by nitrogen, oxygen, carbon monoxide, ammonia, deuteroammonia, methane, deuteromethane, carbon dioxide, as well as
- quantum crystals of the isotopes of hydrogen or helium.

The book demonstrates the experimental data on uniaxial tension or compression strength and ductility, hardness, extrusion, impact toughness, creep, stress relaxation; it also delivers the elasticity characteristics like Young's modulus, shear modulus, Poisson's ratio, and elastic constants. The quantitative data are also given on the thermodynamic characteristics of plastic-deformation processes, lattice imperfections (dislocations, vacancies, and stacking faults), as well as on the effect of isotopic or chemical impurities on the elasticity or plasticity characteristics of the simplest molecular crystals.

The handbook is designed for the wide range of specialists that make use of simplest molecular substances (crystals) both in the research and application areas. It also may be of interest for the scientists, post-graduates, and students working in molecular physics or physics of plasticity, as well as in the engineering areas of spacecraft, high-pressure, or low-temperatures.

FOREWORD

Currently, it becomes common to call *cryocrystals* the simplest molecular substances whose melting occurs well below the room temperature. These are the solidifying inert gases (*rare-gas solids*) and the crystalline bodies formed by diatomic (H_2, N_2, O_2, CO, F_2, *etc.*) or polyatomic (CO_2, N_2O, NH_3, CH_4, *etc.*) molecules. In the molecular cryocrystals different from the rare-gas solids, the lattice interaction pattern depends on not only the intermolecular distance, but also the orientation of the molecules relative one another. It is the strength and kind of the off-center interaction of the molecules that mainly influence the peculiarities of the formation of crystal structure, polymorphism, rotation intensity of the molecules, and thermodynamic or mechanical properties of the crystals.

A crystal lattice of the cryocrystals typically exhibits high symmetry, and the molecular interaction in them is plane and weak. Accordingly, the cryocrystals more than other solids satisfy the favorite theoretical models and provide ideal objects as concerns the investigation into the most critical problems of solid-state physics like those of lattice dynamics, phase transitions, lattice defects, elementary excitations, physics of solid solutions, oriented glasses, *etc.*

Today, the cryocrystals, including their solid-solution forms, are increasingly applied in engineering, particularly, in the

cryogenic or spacecraft equipment (cold accumulators or regenerative expansion systems);

experimental nuclear physics (as the constituents of gamma-radiation gages);

fusion reactors (producing high-temperature plasma with the use of solid deuterium);

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as energy-storing substances or oxidants (solid hydrogen, methane, oxygen, or fluorine);

as the working bodies in ultrahigh-power lasers; or

as the low-temperature conductors of hydrostatic pressure.

To make possible efficient application of the cryocrystals, a comprehensive system of the experimental data on their physical properties is required.

Early as in the 1930s, investigations began into the physical properties of the cryocrystals, including their solid-solution alternatives. Starting from the 1960s, complex systematic studies were conducted of the structure and the thermodynamic, acoustic, and spectroscopic properties. The studies were predominantly done in the Ukraine, in the Physical-Technical Institute of Low Temperatures, National Academy of Sciences (NAS) of the Ukraine, Kharkov Physical-Technical Institute, Donetsk Physical-Technical Institute, and Institute of Physics, NAS of the Ukraine. Likewise, leading physical laboratories of Russia, USA, Great Great Britain, Germany, Japan, Canada, *etc.* performed the investigations. The investigation results were published in hundreds of scientific papers and generalized and discussed in monographs and handbooks:

Rare Gas Solids, Klein, M. L. and Venables, J. A., Eds., Acad. Press, London–New York–San Francisco, 1977;

Cryocrystals, Verkin, B. I. and Prikhot'ko, A. F., Eds., Naukova Dumka, Kiev, 1983;

Physics of Cryocrystals, Manzhelii, V. G. and Freiman, Yu. A., Eds., AIP, New York, 1996;

Roder, H. M., *et al.*, Survey of the Properties of the Hydrogen Isotopes below their Critical Temperatures, NBS, *Technical Note 64 1*, U. S. Government Printing Office, Washington, DC 20401, 1973;

Eselson, B. N., *et al.*, *Properties of Liquid and Solid Helium*, Standarty Publ. Co., Moscow, 1978;

Eselson, B. N., *et al.*, *Properties of Liquid and Solid Helium: ^3He–^4He mixtures*, Naukova Dumka, Kiev, 1982;

Verkin, B. I., *et al.*, *Handbook of Properties of Condensed Phases of Hydrogen and Oxygen*, Hemisphere, New York, 1991;

Manzhelii, V. G., *et al.*, *Binary Solution of Cryocrystals*, Begell House, New York, 1996;

Manzhelii, V. G., *et al.*, *Structure and Thermodynamic Properties of Cryocrystals, Begell House*, New York, 1998;

and other publications.

However, the data on the mechanical properties, including elastic characteristics, are quite scarce in those books.

In the present handbook, the literature data are generalized and critically assessed on the characteristics of mechanical behavior (the strength and ductility values under, compression, tension, impact bending, extrusion, indentation,

creep, *etc.*) and elastic properties (Young's modulus, Poisson's ratio, and elastic constants) of the most plane molecular crystals. The book involves the data published until 1998. Plus to this information, the data are given on the dislocation density, dislocation mechanisms, and thermal-activation characteristics (activation energy and activation volume) of plastic deformation of the cryocrystals. Quite often, nonstandard techniques or facilities were developed and used by the researchers for growing or testing of the specimens. Therefore, we describe some of those techniques and facilities in the handbook. We believe hope that thereby we help the users of the book to apply some of those technical solutions in the professional activity and, as well, to facilitate the appreciation of the delivered data itself.

Compiling the handbook, we partly adopted the information on the plasticity and elasticity of the cryocrystals involved in the above monographs. Yet it is original works that the general piece of the data was taken from. We basically give the quantitative experimental data in a tabulated form. And we use graphs (figures) to represent in general the cryocrystal strength, ductility, or elasticity as dependent of various physical parameters.

The author is humbly and with buoyant feelings dedicating this piece of work to the memory of his parents, Ivan Akimovich Prokhvatilov and Efrosin'ya Maksimovna Prokhvatilova, as well as of Academician Boris Ieremievich Verkin, the founder of the Khar'kov Physical-Technical Institute of Low Temperatures, Academy of Sciences of the Ukraine. Academician B. I. Verkin permanently paid his lively attention to and supported by all means the studies into the physics of the cryocrystals, which were being conducted in the Institute.

I also am sincerely grateful to L. A. Alekseeva, Yu. E. Stetsenko, Candidates of Physical and Mathematical Sciences, and to my dear wife, N. V. Prokhvatiliva, whose assistance and valuable recommendations helped me permanently in the preparation of the manuscript.

NOMENCLATURE

σ_b, σ_τ ultimate (nominal) stress or yield stress, respectively;

σ_s ultimate shear strength;

$\varepsilon_b, \varepsilon_f$ strain values at ultimate (nominal) strength or fracture stress, respectively;

v_d cross-head speed (tension or compression) of the test machine;

ε^* creep-strain rate (creep rate);

ε_0^* diffusion-controlled flow strain in solid helium;

v_l, v_t longitudinal or transverse sound speed, respectively, in a crystal;

a, b, c crystal lattice parameters;

b Burgers vector;

ρ' locked dislocation density;

ρ_0 mobile dislocation density;

C_{ij} elastic constants;

B_T, B_S isothermal or adiabatic elastic modulus, respectively;

E_S, G_S adiabatic Young's modulus or adiabatic shear modulus, respectively;

E_c, G_c static Young's modulus or static shear modulus, respectively;

T_m melting temperature;

η^* solid-state viscosity;

ρ substance density;

U activation energy of plastic deformation of a crystal;

U_0 energy barrier for dislocation motion in a crystal;

γ activation volume of plastic deformation of a crystal;

τ, τ_0, τ_f current, initial, or limiting stress, respectively, in a crystal under stress-relaxation condition;

ξ width of dislocation;

ζ	stacking-fault energy;
ξ^*, s^*	stress-relaxation constants;
H	hardness;
ν_0	oscillation frequency of atoms or molecules;
a_k	impact toughness;
f	frequency of torsion oscillations;
Δ	low-frequency damping;
Q^{-1}	internal friction;
σ_L, ε_L	potential constants of atomic (molecular) interaction;
M	work-hardening coefficient of crystals;
$\lambda_t, \lambda_\varphi$	De Boer's parameter of translation or libration oscillations, respectively;
h	Planck's constant;
h_0	indentation depth (hardness measurements);
α_0	logarithmic-creep constant of crystalline materials;
β	exponential-creep coefficient of crystalline materials;
m	strain exponent for high-temperature creep of polycrystalline materials.

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Monoatomic Crystals (Rare Gas Solids)

Inert-gas crystals belong to the number of simplest solids. They consist of spherical chemically neutral atoms, whose interaction power, determined by the weak van der Waals forces, only depends on the atomic spacing. A lot of their properties can be well described with the use of Lennard-Jones potential of 6–12 pair interaction,

$$\varphi(r) = 4\varepsilon_L((\sigma_L/r)^{12} - (\sigma_L/r)^6) \qquad (1.1)$$

where ε_L is the interaction energy (potential well), σ_L is the atomic diameter, and r, the equilibrium atomic spacing.

In their monograph, Klein and Venables (1977) critically assessed the vast information, published by 1977, about the properties of the rare gas solids. The authors discussed the up-to-date knowledge of the elastic properties of the rare gas solids; yet they quite briefly touched the data on plastic deformation of solid argon only.

Some of the structural and thermodynamic characteristics of the rare gas solids are given in Table 1.1. In the Table, we quoted the properties revealed by the substances of purity as high as 99.95 to 99.99% and in the units employed in the respective original studies.

Table 1.1 The general structural and thermodynamic characteristics of the rare gas solids

Prorerties	Substances			
	Argon Ar	Krypton Kr	Xenon Xe	Neon Ne
Atomic weight	39.95	83.80	131.30	20.18
Triple point				
T_m, K	83.78	115.76	161.39	24.56
P_m, hPa	688	730	816	433.5
Structure type	face-centered cubic (*fcc*)	face-centered cubic (*fcc*)	face-centered cubic (*fcc*)	face-centered cubic (*fcc*)
Lattice parameter at the absolute zero, E	5.311	5.646	6.132	4.464
Parameters of the Lennard-Jones				
σ_L, E	3.405	3.624	3.921	2.788
ε_L, K	119.8	164.0	230.4	36.7
Nearest-neighbor distance, E	3.755	3.992	3.921	3.156
Heat of evaporation, J/mole	6450	9055	12600	1735
Heat of fusion, J/mole	1182	1642	2266	329.4
Molar volume, cm³/mole	22.415	26.932	34.73	13.31
Debye temperature at the absolute zero, K	93.3	71.9	64.0	75.1

1.1 Argon

1.1.1 Active Uniaxial Tension

Stensfild (1956) was the first to examine plastic deformation of flat open specimens of solid argon subjected to active uniaxial tension. Stensfild developed a smart technique of growing the argon specimens free of contact with the walls of the cap-sule and used it to study into a specimen deformation. Argon specimens (99.98% was the initial gas purity) grew to 3–4 mm per hour from the liquid phase in a rectangular glass capsule with its bottom part expanded like a snake's

head. Thanks to a rectangular form of the capsule, it was not particularly a problem to also keep examined the specimen surface with the use of an optical microscope before as during deformation. A tension rod was attached to the test specimens by freezing an anchor-ended thin-wall tube into the top part (also expanded) of a specimen. Thus prepared specimens were transparent, with a polycrystalline structure of about 2-mm grain size. Heating to a near-melting temperature and evacuation of the capsule interior allowed one to untie the specimen from the capsule walls. At that temperature, extensive selective sublimation occurred at the solid-argon/glass interface thanks to a high partial pressure of vapor above the solid argon body. In 20 to 30-minute time, about 30-mm long portion, i.e. the gage length, of the test specimen became disconnected from the glass wall. Such kind of thermal-vacuum etching revealed as well the grain boundaries and, under favorable conditions, the exit sites (etch pits) of individual dislocations. With the loose portion thus formed, tensile deformation was possible by loading applied to the pull rod, the bottom end of the specimen fixed within the expanded part of the capsule. This technique made a ground for developing new uniaxial-tension facilities to be involved in the study of crypcrystal plasticity (Verkin and Pustovalov, 1982).

Later on, the plasticity effects of argon became the subject of careful investigations by Bol'shutkina et al. (1972a), Stroilov et al. (1972), and Leonteva et al. (1969, 1980, 1982, and 1983). The starting gas was pure to 99.99%. To perform uni-axial tension, they used an installation, depicted schematically in Fig. 1.1, created based of the modified ideas from Stensfild (1956). A curved round ampoulee served a beaker for growing an argon crystal out of the liquid phase. 1:5 was the gage diameter/length ratio of the tensile specimens. The ampoule would be of 5 or 10 mm in diameter, depending on the peculiarities of a substance to be crystallized. The facility would makes it possible to grow crystals of the most of substances and study into their uniaxial-tension behavior at the deformation rates from 10 to 10^{-5} mm/min and temperatures between 1.5 and 200 K. Using such an installation, all of the hereby-considered substances, save ammonia or helium, were deformed. One can select from two alternative designs of the ampoule bottom to respectively maintain the specimen temperature beyond or below the boiling temperature of the refrigerant. A thermal valve 10, pressure adjusters 19 of the heat-exchange gas (mostly helium) in the jacket, and electrical heaters 8 all round the ampoule make it possible to achieve and maintain a certain temperature level.

To become condensed, a gaseous substance under study would be let through a port 14 into a preliminary chilled ampoule. Then, a crystal specimen would be grown in a temperature–gradient field. The temperature conditions would be selected as to keep the solidification front nearly flat and the growth rates ranging
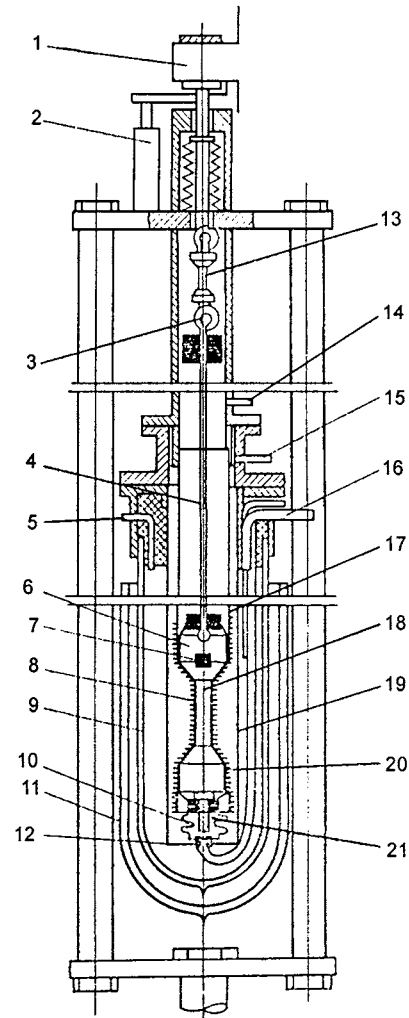
Fig. 1.1 A facility (schematic) for growing specimens of solid-state gases and testing them by uniaxial tension at various temperatures (Verkin and Pustovalov, 1982):

1, immovable part of a loading device; 2, transducer gage; 3, joints; 4, tension rod; 5, drainage tube of the helium vapor; 6, upper grip of the test facility; 7, temperature gage; 8, heaters; 9, helium-chilled Dewar flask; 10, thermal valve; 11, nitrogen-chilled Dewar flask; 12, coolant conduit; 13, dynamometer; 14, inlet for an examined gas; 15, evacuation line of the specimen housing; 17, specimen ampoule; 18, specimen; 19, housing to be evacuated; 20, lower grip; 21, cooland conduit.
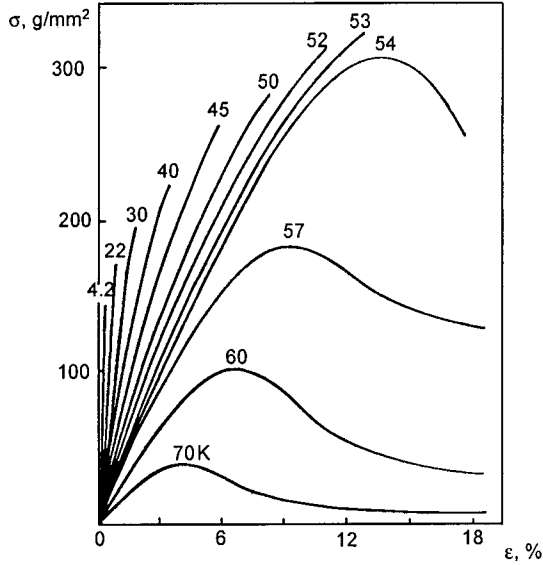
Fig. 1.2 Sress–strain curves of polycrystalline argon subjected to uniaxial tension at the strain rate $v_d = 3.3 \cdot 10^{-4}$ s^{-1} (Leonteva *et al.*, 1988).

from 0.1 to 0.5 mm/min. Thereby, transparent specimens, uniform lengthwise, would usually be obtained, with 0.2- to 1-mm grain size. As in the Stensfild (1956) case, thermal–vacuum etching near the melting temperature ($0.8T_m$ to $0.9T_m$) was used to have the crystal body disconnected from the ampoule walls. The loading stiffness of 2×10^{-3} mm/kg was typical of the as-described test facility, and the measuring errors of applied stress or relative elongation were 4–5% and 1%, respectively. The crystals behavior was examined in the temperature range 4.2 to 77 K at the strain rates of $5.5 \cdot 10^{-4}$ and $5.5 \cdot 10^{-5}$ s^{-1}. Figure 1.2 depicts strain-hardening curves of argon at various temperatures. The curves appear typical of polycrystalline materials. The stress values σ appear proportional to the strain values ε at the early stage of deformation. Yet the proportionality coefficient appears strongly dependent of temperature and an order of the magnitude as small as the Young's modulus E of the argon specimens. Obviously, the linear portion of the stress–strain curves signifies the linear strain-hardening, not elastic, behavior of the crystals, $K = \sigma/\varepsilon$ being the strain hardening coefficient. The linear portion terminates at the point conventionally defined as an engineering (nominal) yield stress σ_τ. A stage of parabolic stress–strain relationship follows the linear portion of the curve until the greatest nominal stress is achieved, called

Table 1.2 Uniaxial tensile properties: ultimate tensile stress σ_b, yield stress σ_τ, relative elongation ε_b, corresponding to the ultimate tensile stress, and relative elongation to fracture ε_f of polycrystalline argon at the strain rate $v_d = 3.3 \cdot 10^{-4}$ s^{-1} (Bol'shutkin et al., 1972a).

Properties	Temperature, K							
	5	10	15	20	25	30	35	40
σ_b, g/mm^2	160	160	160	159	157	155	154	150
σ_τ, g/mm^2	151	136	121	107	95	82	71	61
ε_b, %	0.6	0.8	1.0	1.1	1.2	1.4	1.6	2.5
ε_f, %	0.6	0.8	1.0	1.1	1.2	1.4	1.6	2.5
	45	50	55	60	65	70	75	
σ_b, g/mm^2	144	130	116	99	80	60	38	
σ_τ, g/mm^2	54	49	44	43	40	30	20	
ε_b, %	5.3	9.0	11.8	10.0	9.2	7.0	4.0	
ε_f, %	5.3	9.0	14.0	19.0	23.0	26.0	30.0	

ultimate tensile stress, σ_b[†]. At low temperature, fracture of the material normally occurs as soon as the ultimate tensile stress is achieved. Yet at higher temperatures, applied stress is likely to diminish gradually from the maximum down to fracture-stress level (nominal) according to a gradual significant reduction (local necking) of the specimen diameter (Fig. 1.2).

There are two temperature ranges distinctly different as concerns the deformation behavior or fracture appearance of argon crystals. In the low-temperature range ($T < 53–55$ K), the specimens would deform uniformly all over the gage length, and fracture occur once the σ_b level was achieved. In the higher-temperature range, deformation would be localized in one or several necks at $\varepsilon > \varepsilon_b$, and the crystal would exhibit ductile fracture.

The strength and ductility characteristics given in Table 1.2 were obtained based on the analysis of the deformation curves of argon crystals in the temperature range 5 to 75 K. In Figs. 1.3 to 1.5, the magnitudes of ultimate tensile stress

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[†]In the Russian engineering language, this stress level (*nominal tensile strength*) is referred to as *strength limit or temporary resistance to rupture*, conventionally labeled as σ_B. In fact, index B should to be transliterated as w (not b!), for Russian *wremennoe*, i.e. English *temporary*. Yet σ_b, though wrong, is widely recognized a subscript. (*Transl.*)
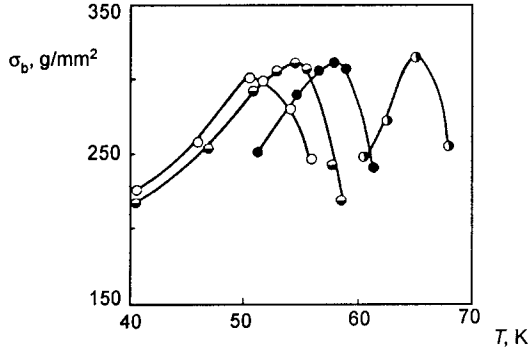
Fig. 1.3 Uniaxial tensile strength σ_b as a function of temperature for polycrystalline argon at the strain rates (O) $1.01 \cdot 10^{-4}$, (◔) $3.3 \cdot 10^{-4}$, (●) $5.5 \cdot 10^{-4}$, or (◑) $5.5 \cdot 10^{-3}$ s^{-1} (Leonteva et al., 1986).
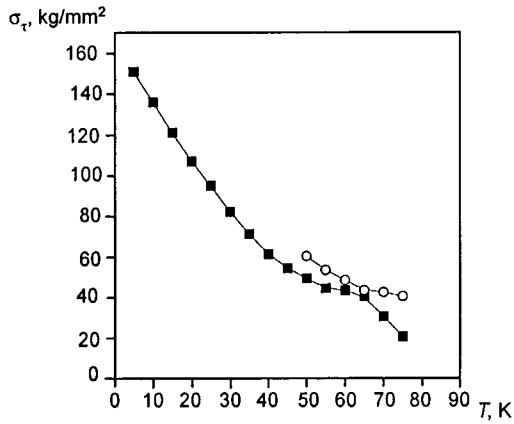
Fig. 1.4 Uniaxial tensile yield strength σ_τ as a function of temperature for polycrystalline argon at the strain rates (■) $5.5 \cdot 10^{-4}$, or (O) $5.5 \cdot 10^{-3}$ s^{-1} (Bol'shutkin et al., 1972a).

σ_b, yield stress σ_τ, (both nominal) or relative fracture elongation ε_f are plotted as functions of temperature. According to the above data, the strength and ductility behaviors of the solid argon obviously alter with changing from the temperature range of brittleness to that of ductility. The brittleness range increases by almost 15 K as the strain rate grows from $1.01 \cdot 10^{-4}$ to $5.5 \cdot 10^{-3}$ s^{-1}(Fig. 1.3). One should mind that the abrupt decrease of the strength and ductility characteristics at the low temperatures (brittle-fracture range) might occur once the specimen surface is damaged due to the thermal-vacuum etching. Kucheryavii and Stetsenko (1971) and Grushko (1974) proposed the methods of making and testing the loose
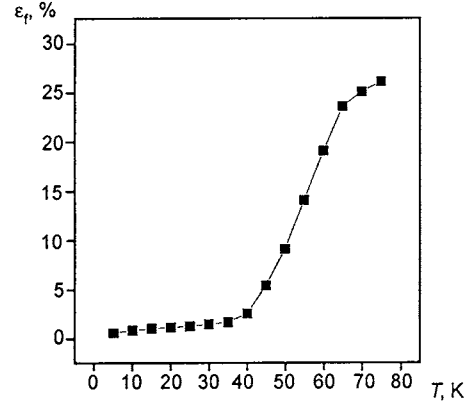
Fig. 1.5 Relative elongation to fracture ε_f as a function of temperature for polycrystalline argon uniaxially stretched at the strain rate $5.5 \cdot 10^{-4}$ s^{-1} (Leonteva *et al.*, 1988).

specimens of solidified gases with yet undamaged smooth surface. However, the methods were never widely applied.

1.1.2 Creep and Stress Relaxation

Creep. Leonteva *et al.* (1980, 1983, and 1985) and Romanusha (1987) examined the creep behavior of argon in the temperature range 4.2 to 77 K. The starting-gas purity was not less than 99.98%. The specimens were grown and tested using the test device (Fig. 1.1) for active uniaxial tension. Yet the loading device was of the balance (instead of reduction-gear) type, with one of the shoulders attached to the upper pull rod, frozen into the test specimen (Fig. 1.1), and the other shoulder, loaded with a constant weight.

Figure 1.6 illustrates the typical creep curves at various temperatures of the temperature range 4.2 to 50 K of embrittlement of argon. Similar to other solids, the argon specimens exhibited the stages of transient and steady-state creep. Leonteva *et al.* (1986) noticed that essentially no plastic flow would occur at the steady-state-creep stage in the range 4.2 and 20 K, i.e. the creep rate was zero.

At the primary creep stage, the creep strain shows itself a logarithmic function of time (Fig. 1.7) in the temperature range 4.2 to 45 K,

$$\varepsilon = \alpha_0 \ln t + C \qquad (1.2)$$

to manifest the dominating role of hardening processes. The time dependence $\varepsilon^* = f(t)$ of creep rate deviates from linearity at 49 K, *i.e* in the vicinity of the
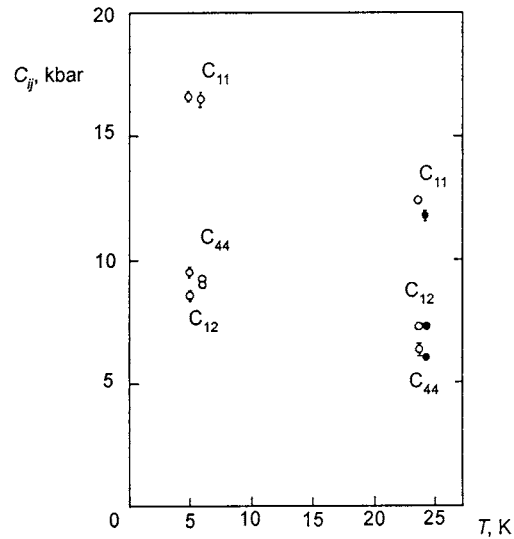
Fig. 1.61 The adiabatic and zero sound elastic constants of solid neon (Klein and Venables, 1977). Open circles: measured with inelastic neutron scattering by Scalyo *et al.* (1972) at 5 K and by Endoh *et al.* (1975) at 6 K and 23.7 K. Full circles: measured with spontaneous Brillouin scattering by McLaren *et al.* (1975) at 24.3 K.

1.3 K. The sound velocities were measures with the use of a phonon-interferometry technique. According to Spiel (1999), the longitudinal sound velocity in the quench-condensed films would be comparable with that in the equilibrium bulk crystals. Yet the transverse sound velocity was almost twice as low as that in the bulk material.

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