

NUCLEAR
ENGINEERING
MONOGRAPHS

NUCLEAR
REACTOR
MATERIALS

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M. B. WALDRON

TEMPLE PRESS



NUCLEAR REACTOR MATERIALS

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TEMPLE PRESS LIMITED
BOWLING GREEN LANE, LONDON E.C.1

(2)

RD 23-14-1

Preface

The previous monographs in this series, dealing with the physics and engineering of nuclear reactors, have outlined the enormous amount of information which has been obtained in these fields over the past fifteen years. However, in order to put physical and engineering theory into practice a wide variety of materials of construction is required: fissile or fertile material in the form of metals or ceramics, clad in metal of low neutron-absorption cross-section, placed in graphite or water moderator (in a thermal reactor) and cooled by a suitable heat transfer medium. The development of such materials with the required physical and mechanical properties in the presence of high neutron and gamma fluxes has been, and still is, a formidable task. Metallurgists in many countries have taken up this challenge. They have developed the techniques for extracting many of the rarer metals, such as beryllium, zirconium and niobium, from their ores and for their fabrication into sheet, tube and bar. Many of their physical and chemical properties have been measured both in the laboratory and in high neutron fluxes. Moreover, it has often proved possible to explain their behaviour in terms of the modern theories of the solid state.

It is essential for the reactor physicist and engineer to appreciate the possibilities and the limitations of the materials with which he has to work. The primary aim of this monograph is to outline for such people the present state of the art and science of reactor materials. To facilitate this explanation the first part of the book deals at some length with some of the more important aspects of modern metallurgical theory, in the course of which a number of common metallurgical terms are introduced. Subsequent chapters deal with fuel, canning and moderator materials while the final chapters are concerned with the problems of corrosion by various coolants of fuel and canning materials.

The monograph is based on a series of lectures by staff of the Metallurgy Division, A.E.R.E., Harwell, to students in the Department of Nuclear Engineering, Queen Mary College, London. The chapter on ceramic materials has been written by Mr. J. Williams, Group Leader of the Ceramics Group at Harwell and the chapter on gaseous and aqueous corrosion by Mr. J. N. Wanklyn, Section Leader of the Corrosion Section at Harwell. Much of the subject-matter is concerned with work carried out in the Metallurgy Division, Harwell, and where possible reference has been made to the published versions of such work from which many of the illustrations are taken.

The authors would like to acknowledge the initiative of Professor W. Murgatroyd and his staff at Queen Mary College in arranging the course on which this book is based and in making many useful suggestions for its subject-matter.

Atomic Energy Research Establishment
Harwell. *June* 1959

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Chapter One

THEORETICAL METALLURGY

INTRODUCTION

Most of the important materials of atomic energy are metals, the notable exceptions being graphite, heavy water and gaseous coolants. Fuel, blanket and structural materials are all based on metals, and the study of the metallurgical problems which they pose is one of the more important branches of reactor technology.

The common structural metals and alloys crystallize in fairly simple atomic arrangements. However, a number of metals used in the nuclear field, particularly the fissile metals uranium and plutonium, crystallize in complex structures. Not only do they have complex structures but they also undergo changes in structure as temperatures are varied. It is therefore necessary to study the different types of crystal structure occurring in metals, alloys and compounds and the way in which these structures are built up into a polycrystalline material.

The physical properties of metals are partly dependent on the properties of the constituent atoms and partly on the arrangement of these atoms. The chemical properties on the other hand are almost entirely dependent on the atomic characteristics. Because physical properties have some dependence on the crystal structure, which can be distorted by deformation, these are said to be "structure sensitive"; chemical properties are, in general, "structure insensitive".

The metallurgical data required by reactor engineers and physicists for design purposes should, ideally, include the following properties:

Physical properties

Crystal structure	Electricity conductivity (ohms/cm^2)
Density (g/cm^3)	Thermal conductivity ($\text{cal/cm}^2 \text{ } ^\circ\text{C sec}$)
Melting point ($^\circ\text{C}$)	Thermal expansion ($\text{cm/cm}^\circ\text{C}$ and $\text{in./in. } ^\circ\text{C}$)
Vapour pressure (mm Hg)	Diffusion constants (cm^2/sec)
Diffusion constants (cm^2/sec)	Specific heat ($\text{cal/g } ^\circ\text{C}$)

Chemical properties

Mechanical properties

Deformation modes	Fatigue properties
Tensile properties	Impact value
Creep properties	Hardness

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Alloying behaviour

Equilibrium diagrams

Microstructure

Corrosion behaviour (compatibility)

All but the last item will be discussed in the remainder of this chapter, together with a brief examination of the effects of irradiation on some of these properties. Corrosion is discussed in Chapters Four and Six.

CRYSTAL STRUCTURE

Atomic Bonding, (see Mansfield (1958) in Bibliography, page 74)

Ionic bonding: Uniform "electrostatic" field.

Covalent bonding: Directed, shared linkages.

Van der Waals' forces: Weak dipole forces.

Metallic bonding: Free electrons give uniform attraction between ions.

Most metals have entirely metallic bonding but a few, e.g. bismuth, have some covalent bonding which is associated with complex crystal structures.

Atomic Arrangements

These are explained in terms of the "unit cell" which is the smallest unit by which the lattice can be described. The 14 basic types of unit cell are shown in Fig. 1 (overleaf).

1. Face-centred cubic (F.C.C.). Atoms at the corners of a cube and in the centre of each face. Each atom surrounded by 12 others at equal distance (i.e., co-ordination number is 12).

Examples: copper, aluminium, silver.

2. Close-packed hexagonal (C.P.H.). Atoms at corners of a hexagonal prism, plus one at centre of end faces and group of three in mid-plane. Each atom surrounded by 12 others at equal distance. The vertical dimension c , divided by the horizontal dimension $a = c/a$ is the "axial ratio" the ideal value of which is 1.63.

Examples: magnesium, zinc, beryllium.

These two forms of crystal represent the closest possible packing of spheres into a repeatable lattice.

3. Body-centred cubic (B.C.C.). Atoms at corners of a cube plus one in the centre of cube. Each atom surrounded by eight others at equal distance.

Examples: sodium, molybdenum, niobium, tantalum.

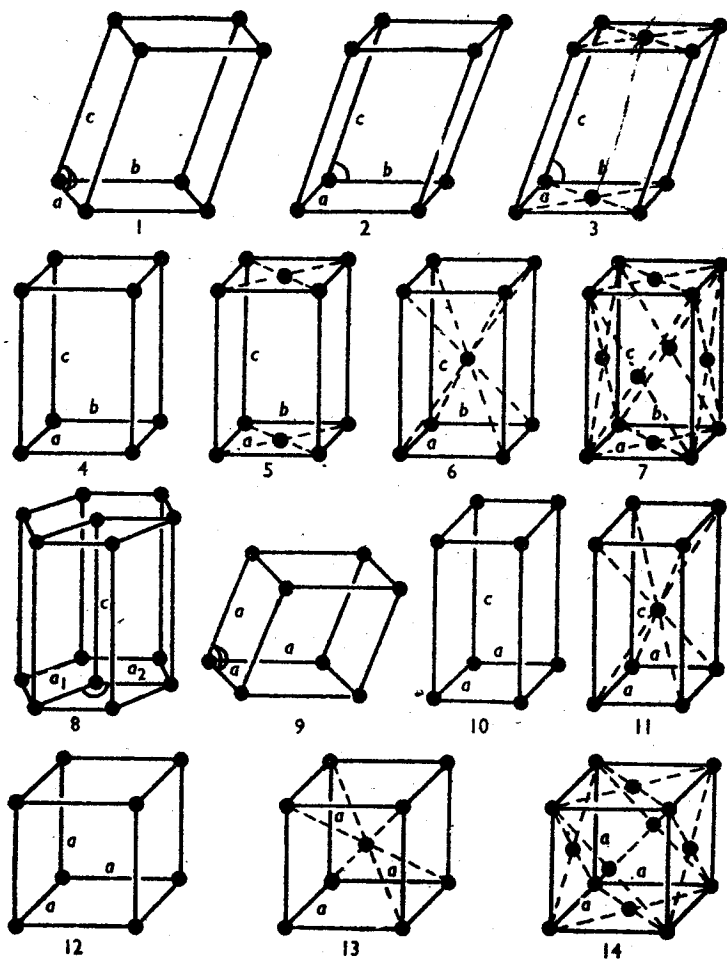


FIG. 1. *The 14 basic types of unit cell*

(1) triclinic, simple; (2) monoclinic, simple; (3) monoclinic, base centred;
 (4) orthorhombic, simple; (5) orthorhombic, base centred; (6) orthorhombic,
 body centred; (7) orthorhombic, face centred; (8) hexagonal; (9) rhombohedral;
 (10) tetragonal, simple; (11) tetragonal, body centred; (12) cubic, simple;
 (13) cubic, body centred; (14) cubic, face centred.

4. Some of the less typically metallic elements have complex structures. Apart from the fissile elements, the principal examples are:

tin: tetragonal,
 bismuth and antimony: rhombohedral,
 gallium: orthorhombic.

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5. A number of elements undergo changes in structure as temperature varies (phase transformation). The best example is iron which is body-centred cubic up to 906°C , face-centred cubic from 906° to $1,403^{\circ}\text{C}$ and body-centred cubic again from $1,403^{\circ}\text{C}$ to the melting point at $1,535^{\circ}\text{C}$. Metals of nuclear importance exhibiting phase transformation are:

Change temperature	from	to
Beryllium: 630°C	Close-packed hexagonal (C.P.H.)	Hexagonal
Zirconium: 862°C	Close-packed hexagonal (C.P.H.)	Body-centred cubic (B.C.C.)
Thorium: $1,400^{\circ}\text{C}$	Face-centred cubic (F.C.C.)	Body-centred cubic (B.C.C.)

6. Uranium has three allotropic forms:

- α : stable to 660°C —orthorhombic,
- β : from 660° to 760°C —tetragonal,
- γ : from 760° to $1,133^{\circ}\text{C}$ —body-centred cubic.

Plutonium has six allotropic forms, these are listed and discussed in Chapter Three.

As will become apparent later the optimum properties are associated with the simple close-packed crystal structures (B.C.C. and F.C.C.) and the object of alloying is to stabilize these structures down to room temperature, i.e. γ -uranium and δ -or ϵ -plutonium. Alloys between two or more metals form *solid-solutions*, in which the original elemental crystal structure remains intact but slightly distorted, and *compounds* when a different crystal structure (often complex) is formed.

Crystallographic Notations

A lattice is made by repeating the "unit cell" in three dimensions. Within a perfect crystal the atomic pattern is continuously repeated and by drawing planes at different angles through the lattice one can intercept a number of atoms. Many of the properties of the crystal are best described in terms of these lattice planes. The direction of the planes are denoted by three co-ordinates known as Miller indices.

Miller Indices. For cubic crystals take three axes at right angles to one another. Then a plane makes intercepts a , b and c with these axes. Any other crystal plane makes intercepts a/h , b/k , c/l where h , k and l are small integers. To specify a plane we write h , k , l and rationalize fractions or reduce to a l.c.m. to give the indices H , K , L , e.g. a plane makes intercepts $2a$, $b/3$, $c/4$ then h , k , $l = \frac{1}{2}, 3, 4$, i.e. Miller indices (168). Brackets () denote one plane and brackets < > denote families of planes. A negative index is $\bar{1}$. Figure 2 shows the indices of some important planes in cubic and hexagonal crystals.

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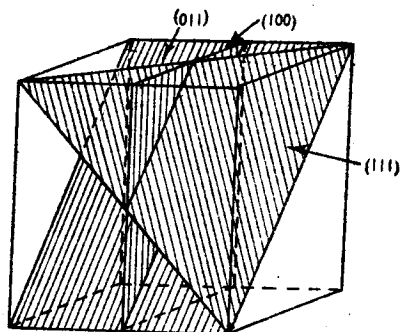
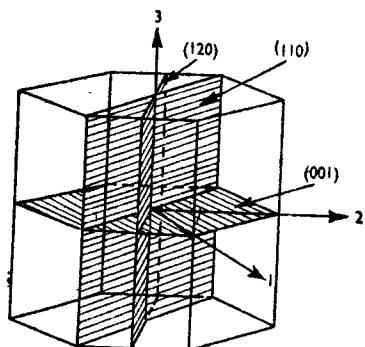


FIG. 2
*Miller indices of planes in cubic
and hexagonal crystals*



A similar notation is used for crystallographic direction using the intercepts of the sides of the unit cell. In *cubic* systems the direction $[hkl]$ is perpendicular to the plane (hkl) , but not in other systems.

Determination of Crystal Structure. In metals the external form of the crystal provides no guide to its structure. The only reliable method is by the diffraction of electron, X-ray or neutron beams by the crystals. A series of planes in a crystal diffracts the beam at a characteristic angle θ such that Bragg's law is satisfied:

$$n\lambda = 2d \sin \theta$$

where n is an integer,

λ is the wavelength of the radiation,

d is the spacing between successive planes.

For the determination of a new structure a single crystal is required. For checking and comparison purposes a powder obtained by filing a polycrystalline material will suffice.

Further details of the above sections will be found in works by Barrett (1943) and Taylor (1949).

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Lattice Defects. In practice a crystal lattice is never perfect. The crystals are composed of a mosaic of blocks tilted slightly ($< 1^\circ$) with respect to one another. Other defects are:

Vacancies—vacant lattice sites

Interstitials—sites between atoms filled by atoms forced from the lattice points.

Dislocations—essentially a region of distortion caused by a unit difference in the number of atoms in adjacent planes (see below).

Vacancies and interstitials are important in relation to structure-sensitive properties. Dislocations explain many features of deformation processes.

The Bulk Material

1. In the commercial form metals and alloys consist of polycrystalline aggregates, the crystals being oriented randomly with respect to one another. In fabrication processes, e.g. wire drawing, rolling and extrusion, the crystals are not randomly oriented; they are usually pulled into the working direction. This is called "preferred orientation" and affects structure-sensitive properties. An alternative description is rolling or drawing "texture". Due to the alignment of the grains properties are highly directional.

2. Grain size is very important. A fine-grained random structure is most desirable for strength and for anisotropic properties. Large grains are produced in slowly-cooled castings and when metal is held at high temperatures for long periods. Alloying elements and impurities can assist in checking grain growth.

DENSITY

This is related to the atomic weight of the element and to the structure in which it crystallizes. Values vary from lithium (atomic number 3, B.C.C., $d = 0.53 \text{ g/cm}^3$) to osmium (atomic number 76, C.P.H., $d = 22.5 \text{ g/cm}^3$). In nuclear engineering one is concerned with the whole range of values from lithium to uranium ($d = 18.5$) and plutonium ($d = 19.7$) including beryllium (1.8), zirconium (6.5) and niobium (8.4).

MELTING POINT AND VAPOUR PRESSURE

The relationship between these properties and atomic properties is not known with clarity. It is sufficient to say that no metals melting below 600°C are of interest as structural materials—these are of primary interest as liquid-metal coolants and fuels. Most structural metals melt above $1,000^\circ\text{C}$, exceptions being magnesium (650°C) and aluminium (660°C). Generally vapour pressures must be low in any application, preferably less than 1 micron (0.001 mm Hg).

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DIFFUSION

The rate at which atoms diffuse through a metal lattice is of considerable importance since it determines the rate at which alloying or contamination by impurities can occur. The diffusion coefficient D is defined in terms of the amount dm of a substance B diffusing in time dt across a surface area A lying between two planes dx apart and differing in concentration dc ,

$$dm = AD \cdot \frac{dc}{dx} \cdot dt.$$

D depends on concentration and on temperature:

$$D = K \cdot \exp(-Q/RT).$$

The activation energy, Q is independent of direction in cubic crystals but not in non-cubic crystals. Diffusion occurs by one of three mechanisms, (i) interchange of atoms in adjacent sites, (ii) movement through interstitial positions, (iii) movement of vacancies. Mechanism (iii) is the most probable. Where two metals are in contact one may diffuse more rapidly into the other and will leave cavities behind—known as the Kirkendall effect.

For physical property data on metals see Smithells (1955).

ELECTRICAL CONDUCTIVITY

The valency electrons in "normal" metals are not bound to atoms but are free to move through the lattice. However, quantum theory postulates discrete energy values for these electrons and these occur in bands of allowed energy values. Only the electrons in top levels can be excited to higher levels and these take part in electrical conduction. In Brioullin zone theory, the band theory is extended in three dimensions in accord with the type of crystal lattice.

In metals the bands or zones are incompletely filled. In ionic solids the zones are exactly filled. In covalent solids there are no free electrons. In semiconductors impurities act as stepping stones to electrons passing to the higher levels.

A perfect lattice should have zero resistance since there is no impedance to the movement of the conduction electrons under an applied field. Resistance is due to lattice vibration—this explains the temperature dependence of resistivity—and to impurities and imperfections in the lattice. Objects of size similar to the electron wavelength (i.e. atomic scale) have a much greater effect than those of larger size; impurity atoms have a greater effect than internal stresses. Values of conductivities vary according to the extent of filling or overlap of Brioullin zones. Monovalent metals are the best conductors. At the

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other end of the scale bismuth has a small zone overlap and is a poor conductor.

THERMAL CONDUCTIVITY

In thermal conduction as with electrical conductivity electron movement is the predominating mechanism and resistance is provided by lattice vibrations and imperfections. The strongly bound metals, e.g. Cu, Ag, Fe, Ni, will be good conductors of heat and electricity since the restoring forces on vibrating atoms will be strong and hence the amplitudes of vibration will be small. In the same way the specific heats of the strongly bound metals will be high.

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Most nuclear metals have non-cubic crystal structures, so electrical and thermal conductivities, diffusion coefficients and coefficients of expansion will vary in the different crystallographic directions. It is important to note that when a change of crystal structure occurs on altering the temperature of a metal, as in uranium for example, the physical properties also change in magnitude and directionality. (See Cottrell (1955).)

CHEMICAL PROPERTIES

Many aspects of the chemical behaviour of the nuclear metals are outside the scope of this particular section and will be considered, e.g. in discussing aqueous and gaseous corrosion. It will suffice at this stage to consider valency and some of the principles of chemical thermodynamics.

Valency

In considering chemical, as opposed to metallurgical and physical behaviour of metals, one is largely concerned with the formation of ionic, and to a much lesser extent covalent, compounds. In forming such compounds with the non-metallic elements such as chlorine, fluorine, hydrogen, nitrogen and oxygen, metals can display varying valencies. It is the electrons in the outer orbitals of atoms which are responsible for binding atoms together or to atoms of another species. These outer or valency electrons are themselves attracted by varying extents to the nucleus and so it is possible for an element to display varying valency, e.g. uranium forms two fluorides UF_4 and UF_6 and is believed to form the oxides UO , UO_2 and UO_3 together with an intermediate form U_2O_8 . These compounds between the same elements but with different valencies differ somewhat in their properties, e.g. the melting points of UF_4 and UF_6 differ by about $1,000^\circ C$.

When an ionic solid dissolves in an electrolyte, e.g. water, it ionizes, i.e. it dissociates into ions of opposite charge, e.g. NaCl splits into Na^+ which is the sodium atom minus a valency electron which it has given to the chlorine atoms to form Cl^- . When the negative ion is hydrogen the electrolyte has acidic properties, when it is OH^- it is alkaline.

In chemical language the exchange of electrons between differently ionized solids (including zero) is referred to as oxidation and reduction, even where oxygen may not be involved, e.g. $\text{UO}_3 + \text{H}_2 \rightleftharpoons \text{UO}_2 + \text{H}_2\text{O}$ is easily recognized as a reduction process in which hydrogen loses one electron/atom and uranium gains two, $\text{UF}_4 + 2\text{CaF}_2 \rightleftharpoons \text{UF}_6 + 2\text{CaF}_2$ however, is an oxidation process involving electron exchange.

Thermodynamics

Chemical thermodynamics is a very useful method of describing the relative stabilities of chemical compounds. One can determine, under a certain set of conditions of temperature and pressure, the way in which a compound reacts *theoretically* with another element or compound. No account is taken of the states of dispersion which will in fact determine the *rate* but not the direction of reaction.

Thermodynamic Laws. A material at a given temperature T ($^\circ\text{K}$) in a certain environment will have an *internal energy* E roughly corresponding to the binding energy or a heat of combination of the atoms. It will also have a certain *entropy* S ; this is a quantity which defines the state of aggregation of the atoms. As a structure becomes more disordered S increases, e.g. with increase in temperature. If the probability of an aggregation of atoms assuming a configuration is p then $S = k \log p$ where k is Boltzmann's constant.

In undergoing a change absorbing a quantity of heat dQ , the entropy change

$$dS = dQ/T.$$

In a practical system at constant pressure and volume for a *reversible* reaction.

$$S = dQ/T$$

for an *irreversible* change $S > dQ/T$.

This is virtually the Second Law of Thermodynamics, i.e. for any reaction the total entropy change in the system and its surroundings must be > 0 . In undergoing a change:

$$dE = T.dS - P.dV.$$

We also define the *enthalpy* H of the system as

$$H = E + PV,$$

$$dH = T.dS + V.dP.$$

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At constant pressure and volume

$$dH - T.dS = 0$$

or

$$d(H - T.S) = 0.$$

The quantity $H - TS = G$, the free energy. The system assumes equilibrium at the condition of minimum free energy. Now it is this free energy which determines the relative stabilities of compounds. It has a numerical value of calories/gramme or gramme-molecule.

If we consider the free energies of the metal oxides at constant temperatures we get the following series in ascending order of stability (i.e. CaO most stable):

TABLE 1. FREE ENERGIES OF METALLIC OXIDES

Oxide	Free energy (kcal/g mole O ₂)		Oxide	Free energy (kcal/g mole O ₂)	
	at 1,000°K	at 300°K		at 1,000°K	at 300°K
CaO	-126	-144	TiO	-101	-117
BaO	123.5	140	V ₂ O ₅	—	92
ThO ₂	125.5	140	Nb ₂ O ₅	76	91
MgO	119	136	Na ₂ O	65.5	90
Li ₂ O	112	134	Cr ₂ O ₃	68.5	83
Al ₂ O ₃	112	126.5	H ₂ O	45.5	55
BaO	109	126	NiO	—	52
ZrO ₂	108	124	PbO	29	45.5
UO ₂	109	123.5	Bi ₂ O ₃	23	40
PuO ₂	106	121			

All the values have a negative sign since the formation of an oxide, as with most other compounds, involves evolution of heat so that free energy change or work done on the system is negative. The implication of the above table is that an oxide can only be reduced by an element higher in the table, e.g. uranium is derived from its ores as UO₂ which can be reduced to uranium metal by reaction with an element higher in the series, usually magnesium or calcium. The latter having the most stable oxide is extensively used in metal reduction processes. Similar tables can be constructed for the fluorides, hydrides etc. to determine the relative stabilities of compounds. (See Kubaschewski and Evans (1955).)

MECHANICAL PROPERTIES

Modes of Deformation

Deformation processes are most easily considered by reference to single crystals, adding the complication of the polycrystalline arrangement at a later stage. Deformation occurs by two mechanisms:

1. *Slip*. The planes of atoms, usually the most closely packed,

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slide over one another like playing cards in a pack. At the same time the tension of the crystal causes the planes to rotate in a direction which tends to bring them more in line with the tension. This is illustrated diagrammatically for a C.P.H. metal in Fig. 3. After this process "slip lines" are visible on the surface, not at planar spacings of $\sim 10^{-8}$ cm but 10^{-4} cm apart.

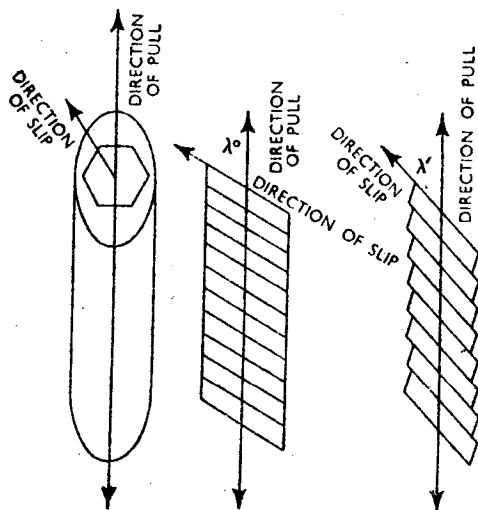


FIG. 3. Slip in a C.P.H. crystal

2. **Twinning.** In this process all the atoms in part of a crystal move in such a way that the whole of the lattice in that area takes up a position which is a mirror image of the remainder, hence the term "twin". This occurs in abrupt steps and can occur under applied stress or as a result of annealing at high temperatures. The deformation of α -uranium at room temperatures occurs largely by twinning.

As deformation by either process occurs the metal becomes progressively harder and less ductile (work hardening) until at very high deformations the crystals begin to fragment.

On heating a cold-worked metal the first process to occur is the relief of stresses within the metal (recovery); this is followed at higher temperatures or after longer times by recrystallization in which new grains grow with different sizes and orientations from the original grains.

Metals deform most easily by movement along the closely packed planes. In F.C.C. and B.C.C. structures there are several closely packed planes. In the C.P.H. structure only the (001) basal plane

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(base of the hexagonal prism) is closely packed so slip directions are restricted. In other structures, with more complex packing and perhaps covalent bonds, deformation is less easy. This does not mean that α -uranium for instance is not ductile, but it is not as ductile as, say aluminium, and its ductility varies in different crystallographic directions. This explains the desirability of stabilizing the high temperature γ -form down to room temperature to give high, isotropic ductility.

When crystals are packed together in a random fashion, as in commercial material, each crystal is trying to deform in a different direction but is restricted by its neighbours and a polycrystalline aggregate has less ductility but greater strength than a single crystal.

Grain Boundaries

Grain boundaries are important for several reasons: (i) they are the region in which the orientation of one grain transforms to that of the next and hence will have an irregular structure which leads to (ii) concentration of defects, and (iii) since they are the last region to solidify they contain a high concentration of impurity atoms. For these reasons the grain boundaries have a marked effect on physical properties and on deformation behaviour.

Dislocations

Many aspects of deformation processes have been successfully explained by postulating the existence of lattice imperfections known

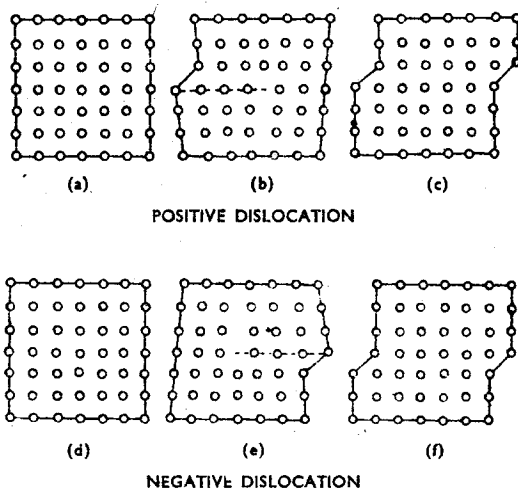


FIG. 4. Dislocations in a simple two-dimensional lattice

as dislocations. The imperfection results from the part of the crystal above the line of the dislocation having one more row of atoms than the part below the line. The centre of the dislocation occurs where the two lattices are half an atomic distance out of phase. The commonest source of dislocations is at the grain boundary. Slip occurs by the movement of this centre along the line to the edge of the crystal, producing a net displacement of the two halves of the crystal relative to one another, as shown in Fig. 4. If a dislocation with an extra row above the line (positive) meets one with the extra row at the bottom (negative) they obliterate one another.

Since dislocations are regions of lattice disturbance foreign atoms tend to locate themselves in the vicinity of dislocations thus restricting their movement. The existence of the yield point in the stress-strain curve has been explained on this basis since the foreign atoms will not permit plastic flow until higher stresses than are needed for pure metals. Once the dislocations are freed of the foreign atoms the stress required drops—hence the yield point.

TENSILE PROPERTIES

Yield Strength. The stress below which all deformation is elastic, i.e. disappears when the stress is removed.

Proof Stress. The stress at which a stated plastic (irrecoverable) deformation occurs, e.g. 0.1 per cent P.S. This is of value where the yield stress is ill defined.

Ultimate Tensile Stress (U.T.S.). The breaking stress.

Elongation to Fracture. A measure of the ductility. This property determines the ability to accommodate local stress concentrations in a structure.

Elastic (Young's) Modulus. The stress coefficient of elastic strain, i.e. stress/strain.

These properties are an important guide to the choice of materials of construction because they are relatively quickly obtained. However, since reactors are designed to operate at elevated temperatures, the tendency to deform plastically under prolonged stress, known as *creep*, is important. The elongation *versus* time curve (at constant load and temperature) has a characteristic shape shown in Fig. 5 in which there is a rapid initial rise (primary creep) merging into a steady rate of rise (secondary creep) and finally curving upwards to fracture (tertiary creep). This behaviour can be interpreted in terms of dislocation theory on the basis that in the primary stage dislocations move under the influence of stress and temperature until they become trapped by impurities, etc. However, at a given temperature a certain number of new dislocations will form at a steady rate and permit steady state deformation. At the tertiary stage either the specimen has so greatly