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Preface

The focus of the thesis is the diffusion along short-circuit diffusion paths in chosen solid Ni-based hi-tech engineering materials.

It is subdivided into following parts: Declaration of *the aim* of the thesis and *the brief survey* of most important results provides the reader with the essence of the work. After the *Short survey of theory*, which offers a short summary of basics needed to put the topic of the thesis into the proper perspective, the *experimental materials* are described. The last part is an overview of *papers published* by the author between the year 1993 and 2003. Most of the published works were carried out in The Institute of Physics of Materials, AS CR Brno, Czech Republic, some of the studies were done at Institut für Metalforschung Westfälische Wilhelms Universität Münster, Germany.

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The aim of the thesis

Grain boundary diffusion is a phenomenon that is controlling for a series of technologically important processes. At the same time, its kinetics is a crucial factor limiting the lifetime of many temperature/stress loaded construction details in machinery. However, quantitative characteristics of the grain boundary diffusion and their temperature and composition dependences are not only important for the practice but they also provide an invaluable insight into the nature of grain boundaries itself that make a ubiquitous structure component of polycrystalline engineering materials.

The aim of this thesis is to obtain grain boundary diffusivities in technologically promising hi-tech materials. The results are interpreted in terms of materials structure and/or interatomic interaction.

Synopsis of the most important results

Ni, Cr and Fe diffusion along grain and sub-grain boundaries in Ni-Fe-Cr austenitic alloys [O5-O8, O11, O13, O14]:

1. Grain boundary diffusivities P of Fe and Cr are approximately two orders of magnitude lower than those for sub-grain boundaries.
2. Grain and sub-grain boundary diffusivities P of Cr are lower or equal to those of Fe.
3. Dependence of activation enthalpies Q_b obtained for Fe and Cr grain boundary diffusion increase with increasing concentration of Cr, whereas the Q_b 's for grain boundary diffusion of Ni show no significant dependence on Cr concentration. This was interpreted by different interaction of diffusing atoms with rest content of carbon and/or by blocking diffusion by fine carbides at grain boundaries. Dependence of Q_b on Ni concentration is not too strong.
4. Traces of phosphorus decrease the grain boundary diffusivity of Fe and increase the activation enthalpy of Fe grain boundary diffusion.
5. Addition of boron to Ni and Ni-9at.%Al decreases the Ni grain boundary diffusivity.
6. Presence of carbon shows the slow-down effect upon the Fe grain boundary diffusion, which is most likely due to Fe – C interaction. The bulk diffusion of carbon itself in Fe-19 at.%Ni-11at.%Cr is accelerated by increasing carbon concentration ($c_C \in 0.01 - 0.81$ at. % C) and it is slowed-down by increasing concentration of both Cr and Fe in Ni-Fe-Cr alloys ($c_{Cr} = 32$ at. % Cr; $c_{Fe} \in 0 - 70$ at. % Fe). The latter effect can be interpreted by the attractive interaction between respective diffusant on one side and carbon on the other. At about $Ni_3(Cr,Fe)$, there was observed a slight tendency to atomic order (extremes in Arrhenius parameters).

Grain boundary diffusion of carbide forming elements Mo, V and W in ferritic Fe-Cr alloys [O20, O21]:

1. The observed overall activation enthalpies of grain boundary diffusivity of all three elements are most likely composed of activation enthalpy of grain boundary diffusion itself and of additive (temperature dependent) term coming from the trapping effect of carbides.
2. There is a breakpoint on the Arrhenius diagram of Mo and W grain boundary diffusivity, which coincides most likely with α/α +carbide phase equilibrium temperature. Above this temperature, the grain boundary diffusivity of W is faster than that of Mo and V.

3. Grain boundary diffusivity of Cr, Mo, W and V in binary Fe-Cr alloys is significantly higher than in commercial P91-type Cr steel (8.36 Cr, 0.09 C, 0.56 Mn, 0.20 Si, 0.02 P, 0.01 S, 0.46 Ni, 0.89 Mo, 0.05 Cu, 0.007 Al, 0.20 V, 0.06 Nb and 0.065 N – all in wt. %). The likely explanation is the interaction of diffusants with constituents of the complex P91 alloy.

Grain boundary diffusion in L1₂ intermetallics [O14, O22, O25, O27-O29, O31]:

1. Microalloying with boron decreases the grain boundary self-diffusivity P of nickel, which may be caused by a structure relaxation of grain boundaries in boronized alloys.
2. Values of P for Ni grain boundary diffusion in boronized (0.106 and 0.268 at. % B) Ni₃Al alloys show minimum at stoichiometry, whereas measured values of P for pure Ni₃Al have in stoichiometry its maximum at higher temperatures and minimum at lower temperatures.
3. Increasing concentration of Fe, Cr and Zr in Ni₃Al decreases the grain boundary diffusivity P of Ni, and increases the activation enthalpy Q_b and pre-exponential factor P_0 . The most expressive effect shows Zr, the moderate only effect shows Fe. Grain boundary energy of Ni₃Al-M (M = Fe, Cr, Zr) calculated from Borisov's relation decreases with increasing concentration of M.
4. Values Q_b of Cr grain boundary diffusion are increased by addition of Zr, whereas alloying Ni₃Al with Fe and Cr brings about no significant changes in Q_b for Cr.
5. Values Q_b of Fe grain boundary diffusion are decreased by addition of Zr, whereas alloying Ni₃Al with Fe and Cr brings about no significant changes in Q_b for Fe.
6. The observations sub items 3-5 above invoke an idea that Ni atoms on their diffusion motion in grain boundary of Ni₃Al-M pass location occupied by all modifying elements (Cr, Fe, Zr), diffusing Cr atoms need to visit locations occupied by Zr atoms and diffusing Fe atoms avoid positions occupied by Zr atoms. This behavior may be due to different affinity of diffusing elements to alloy constituents.
7. The grain boundary diffusivity of Ni and Ga in Ni₃Ga is about the same. This supports an idea about relative *elasticity* of Ga atoms, known from literature, which facilitates the motion of large Ga atoms in the grain boundary.

Short notes related to subject investigated

Introduction

Diffusion was recognized as a very basic phenomenon controlling many natural processes. The research in the field has been started many years ago – the two Fick's laws were written down before 150 years and the Einstein's random-walk theory was published some 100 years ago. Since those times, diffusion was constituted as a distinct area of materials science [1-8].

The diffusion can be understood in the frame of classical phenomenology – as it was the case at early stages of research – or as a kinetic one, running at the microscopic level. From the point of view of phenomenology, diffusion may be characterized simply as a mixing of mater, or as a process of approaching the thermodynamic equilibrium. The atomistic aspect of diffusion respects the discontinuous structure of matter and offers the interconnecting links between macroscopic and microscopic characteristics of diffusion.

A surprisingly wide class of kinetic phenomena involving transmission of electric charge, energy and momentum over macroscopic distances may be explained satisfactorily within the diffusion theory. Phase transformations, creep, oxidation, precipitation and solution of the second phase, powder metallurgy, preparation, treatment and application of nano-structured materials may be given just to exemplify the controlling role of diffusion in the field of materials science.

Tracer diffusion

Let us consider an n -component alloy that is in the thermodynamic equilibrium. If one of the components, let us say the i -th, consists of a spatially non-uniform mixture of two isotopes, progressive balancing of the isotope composition occurs in the volume of the alloy. This process may be monitored by physical methods (mass spectroscopy of local samples, radioisotopic techniques if radiotracers are used, etc.). No chemical changes can be observed – the alloy system behaves as being in thermodynamic equilibrium all that time. Under such conditions the general equations describing the transport of matter can be simplified and, hence, the diffusion of the i -th component can be described by the Fick's laws in classical form (gradients of concentration being the driving forces of diffusion). The tracer diffusion coefficient in Fick's laws quantifies the diffusivity D of one component i in a homogeneous alloy; it is sometimes called coefficient of tracer self-diffusion of i -th component since it characterizes the behavior of the component in the thermodynamic equilibrium. Since the diffusion is a typical thermally-activated process, the temperature dependence of D is governed by the Arrhenius equation.

Throughout the thesis, exclusively *tracer self- and/or heterodiffusion of one component* will be mentioned.

Experimental study of diffusion

There is a number of experimental methods and arrangements used for diffusion measurements – see, e.g., the reviews in [1,6,8-11]. Those used in papers involved in the present thesis are the following ones:

Experimental arrangement

Constant source

This arrangement is realized when the constant concentration, c_0 , of diffusing element is maintained at the surface - for instance by the diffusion from a gas-phase with constant partial pressure of diffusion element.

Instantaneous source

The second type of experimental arrangement is realized, e.g., if the diffusing element is deposited in a thin surface layer onto free surface before the diffusion annealing (electrodeposition, vacuum deposition).

Sectioning methods

Their principle consists in subsequent subdivision (*sectioning*) the diffusion zone in plan-parallel layers and in measuring either the content of diffusing element in individual sections (serial sectioning method) or in measuring the rest amount of diffusing element in the remaining part of the sample yet not sectioned (being combined with the tracer method of concentration measurement – residual activity method). Data obtained by residual activity method can be transformed to the common form of concentration – penetration curves provided the attenuation coefficient is known.

Diffusion in ordered alloys

Ordered alloys are two- or polycomponent materials of the type metal/metal or metal/metalloid that can be prepared in certain composition only. This is due to the fact that their structure is atomically ordered. They behave as distinct chemicals - their properties are often significantly different from properties of pure components, therefore, they are called intermetallic *compounds*. The most frequently encountered structures of technologically important ordered alloys are B2, L1₂, D0₃, D0₁₉, L1₀, and complex cubic and hexagonal structures (by A₂B-type Laves phases).

Ordered alloys are in the center of interest for past decades because of their extremely advantageous properties. Some of them are applied as functional materials, as for example Nb₃Sn

(semiconductor), NiTi (shape memory alloy), Ni₃Fe (high permeability material), Co₅Sm (permanent magnets), some of them, in turn, are applied as structural materials (Ni₃Al-X, TiAl).

Differences between diffusion in atomically ordered and disordered states were observed years ago. Empirical conclusions drawn from early experiments may be summarized in following three relations:

- the diffusion rate in the ordered state is typically lower compared to that the disordered state of the same (similar) matrix,
- activation energy in ordered state is usually higher than in the disordered state,
- for A₃B the so called *Cu₃Au – rule* $D^A > D^B$ holds.

As diffusion mediators in atomically ordered (binary) alloys may serve

- vacancies in sites A,
- vacancies in sites B,
- atoms A in sites B and
- atoms B in sites A.
- triple defects (combinations of two vacancies on A or B sublattice + one antisite atom A or B),
- bound pairs of antisites A+B,
- bound pairs of A or B type vacancy + A or B antisite atom,
- split interstitials (dumbbell-like formation of two atoms around one lattice or interstitial position).

As for the diffusion mechanism, it has to be born in mind that the diffusion process in ordered alloys should not decrease *steadily* the degree of order (short-time local decrease of atomic order may be accommodated by a surrounding lattice). It is obvious that this condition is a limitation for possible diffusion mechanisms.

There are several special mechanisms of diffusion on ordered structure.

- (Antisite) vacancy mechanism
- Six jump diffusion mechanism
- Antisite bridge mechanism
- Triple defect mechanism(s)

Grain boundary diffusion

Inorganic one-phase solids that are constituted by not too rapid solidification of the melt consist of many small crystals - grains. Between the grains there are interfacial regions – grain

boundaries – which thickness is about 0.5 nm where the lattice of the one grain is matched with that of the adjoining grain. These grain boundaries are understood as 2D defects in the bulk of the material. It is obvious that these transition regions possess less crystallographic symmetry; their structure is relatively open, and hence, the diffusion in grain boundaries runs very rapidly. The highest diffusion rate can be observed along the triple junctions (lines where three grains are joined) that form a 3D line net in the solid.

The mean grain size spans over a wide range from the order of millimeters down to order of 10^{-9} m by nano-scaled materials.

Inside a single grain, there are other defects: subgrain boundaries, twin boundaries antiphase boundaries in ordered alloys (2D defects) and dislocations (1D defects).

Structure of Grain boundaries

In early studies, the grain boundaries were understood as slabs, which structure was very close to that of a liquid. This simple idea of homogeneous and isotropic interior of the grain boundary is frequently used up to now as an acceptable approximation of the diffusion problem in frame of phenomenological approach. However, the diffusion measurements have shown that it is inevitable to consider certain – surprisingly high – crystallographic symmetry of grain boundaries: (i) the diffusion anisotropy and (ii) relatively complex dependence of diffusivity on the type and orientation of grain boundary was revealed. It was shown in a number of studies that the grain boundary is composed of a relatively small number of elementary structural units in the form of compact polyhedra varied in size that can be combined periodically in certain patterns.

Grain boundaries in ordered alloys

Grain boundaries in ordered alloys show certain specific features, which can be summarized in following items:

- Strong tendency to atomic order (Ni_3Al) implies that the bulk of adjoining grains remains ordered up to very grain boundary core, but it keeps the grain boundary structure relatively open (there is large free volume in the core); chemical composition of such grain boundaries is close to that in the bulk.
- Weak ordering (Cu_3Au) does not sustain the order up to the grain boundary core and, therefore, the chemical composition of the grain boundary differs from the bulk composition. This decrease in the degree of order in the grain boundary core is balanced by a partial relaxation of grain boundary free volume.
- The tendency to sustain the atomic order on one hand and the structural relaxation of the grain boundary core on the other, may not lead to a unique optimal grain boundary – it was

reported that there may exist two structures differing very slightly only in composition and grain boundary energy.

Mechanism of grain boundary diffusion

It is commonly accepted that the *vacancy mechanism* is most likely one for grain boundary diffusion. This is due to published results on pressure dependence of grain boundary diffusion coefficient D_b and on measured values of isotope effect.

Conclusions leading to the idea about well localized vacancy and single vacancy-atom jumps in grain boundary commented above are, however, somewhat revised in latest works of Mishin and co-workers. They carried out a numeric simulation study of grain boundary diffusion in Cu. Using a combination of several numeric methods (molecular statics, molecular dynamics and kinetic MC) they have concluded that the vacancy show significant *delocalization* at certain grain boundary positions. At the same time, they found that the vacancy move both by simple vacancy-atom exchanges and by long jumps *involving several atoms*. The authors also indicated the *interstitial* (interstitial \rightarrow interstitial jumps; other atoms remaining at their positions) and *interstitialcy* mechanisms (interstitial \rightarrow grain boundary lattice site \rightarrow interstitial).

In works by Mishin et al., also more complex mechanisms of grain boundary diffusion in Cu were found: It was shown that even a *concerted motion* or a *ring motion* involving 3-4 atoms is possible.

Mathematical solution to grain boundary diffusion problem

The first solution to grain boundary diffusion problem was published by Fisher [12]. It was an approximate one for a constant source derived under some restrictive presumptions.

An exact solution for a constant diffusion source was found by Whipple [13] for constant source and by Suzuoka [14] for instantaneous source.

LeClaire [15] used both exact solutions to derivation of the useful approximate formulas enabling the easy evaluation GB diffusion characteristics from measurements.

In polycrystals, the diffusion kinetics can be classified into three types after Harrison [16], or into seven types after Mishin and Razumovskii [8]. Reversely, polycrystals can be – with regard to their diffusion behavior – classified into eight distinct classes. Diffusion along moving GB's and along asymmetric GB's was mathematically treated in [17],[18].

Segregation to grain boundaries

The Fisher's model of grain boundary does not include the segregation effects that may occur at grain boundaries. Namely, if there is a difference between the binding free enthalpies of

diffusing atom to the lattice and to the grain boundary, the diffusing atoms tend to segregate to grain boundary.

Due to segregation, the grain boundary width δ should be replaced with $s\delta$, where s is segregation factor – i.e. the ratio of concentration in the GB and in the bulk, close to GB. The value of s may not be identified with the enhancement factor calculated in the frame of equilibrium segregation theory. As a consequence, the grain boundary diffusivity is a *triple product* of segregation factor, grain boundary width and grain boundary diffusion coefficient. Its temperature dependence is – similar to bulk diffusion – governed by Arrhenius equation with pre-exponential factor P_0 and activation enthalpy $H^p = H^b - |H^s|$ (H^b – activation enthalpy of diffusion in grain boundary).

Such a treatment of the influence of segregation on the grain boundary diffusion respects merely the *explicit* effect of segregated (excess) atoms at grain boundary upon the triple product through the value of the segregation factor s . Influence of the changed vibration states at grain boundary and changed population of different grain boundary sites due to grain boundary segregation on diffusivity in grain boundary remains fully ignored.

Experimental materials

Ni and austenitic alloys containing nickel [O1-O14]

Pure nickel is usually not applied as structural material in the technological practice. Its recrystallization temperature by which the cold worked nickel loses rapidly its hardness is about 400°C, which is very low for engineering applications. Ni is used as a reference material in this thesis.

Alloys with nickel as a major component are numerous and there are many applications of nickel-chromium alloys both as structural materials, heating elements and – in some cases – as functional materials (thermocouples). Their principal features are their resistance to heat, oxidation and corrosion and good strength at moderately elevated temperatures.

Nickel is unique among elements added to iron since it forms a continuous series of solid solutions with negligible solidification interval at any composition. In steels, nickel plays an austenite-stabilizing role and, also, it improves the fabricating characteristics and supports the resistivity of the alloy in reactive environments. The most widely spread Ni-containing alloys are austenitic stainless steels (typical base composition: 18 % Cr, 8 % Ni). They are designed for applications at higher temperatures and to specific situations where severe corrosion conditions occur (boiler tubes, superheaters, reheaters, etc.).

There is a huge number of measurements in the literature on bulk diffusion in these materials. Data on grain boundary diffusion, on the other hand, are scarce and are available for some chosen alloys only.

Ferritic chromium alloys [O15-O21]

The slow-down in the use of nuclear power generation is leading to a worldwide interest in the development of more efficient environment-friendly steam turbine plant with high temperature of inlet steam. For a series of the construction parts of the plant (pipework, casings, blades), more advanced materials are available from the gas-turbine technology (nickel-chromium superalloys). However, manufacturing the large units like turbine rotors of these alloys exceeds the current level of technological capability and, therefore, they have to be made of ferritic steels. The optimization of material for these applications has led to 9-12%Cr steels.

Since the structural stability of the material is given, before all, by the growth/dissolution kinetics of carbides, it is obvious that the knowledge of parameters quantifying the diffusion kinetics is crucial for the aimed design and composition optimization of Fe-Cr alloys for power generators of the new generation.

Ni-based intermetallics with L1₂ structure [O22-O34]

Ni-based intermetallics with L1₂ structure are promising materials for many technical applications. Ni₃Al, which is most frequently studied compound, is prominent example of this class. For diffusion studies, also Ni₃Ga is of considerable interest, since diffusivities of both components can be measured.

Polycrystalline Ni₃Al is very brittle, low ductile and it fails by intercrystalline fracture. Moreover, it exhibits a strong environmental sensitivity, connected with the presence of grain boundaries. Their low strength limits the practical use of the compound. A lot of papers was published, dealing with the possible ways to improved ductility.

Grain boundary diffusion in this class of materials is a hot topic in recent years since it may answer many open questions connected with grain-boundary-related material characteristics.

Miscellanea [O35-O36]

Magnesium

Magnesium is a basic component of a series of special light alloys used for numerous applications in a range of the various branches, such as space, aircraft, communication engineering and other applications. The major driving force for development is the automobile industry, which has renewed interest in Mg alloys for improving fuel efficiency through vehicle mass reduction.

Due to low melting point of Mg and Mg alloys, all principal changes involved in technology operations are more or less influenced by diffusion of basic and/or additive chemical components (i.e., micro-alloying elements with ≤ 1 wt.%). Therefore, it is a little bit astonishing that there are relative few data sets describing quantitatively diffusion behavior of Mg alloys.

Grain boundary diffusion of Cu in Cu-Al

Cu-Al alloy is a suitable model for the study of interrelations between characteristics of grain boundary diffusion and Coble creep occurring at relative low stresses σ ($\sigma < 5 \times 10^{-4} G$; G – shear modulus) and low temperatures T ($T \sim T_m/2$; T_m – melting temperature).

Interphase self-diffusion diffusion in Cu-Fe alloy

Cu-Fe heterogeneous alloy was chosen as a model of two-phase media with non-coherent interphase boundaries, with significantly different bulk diffusion coefficients in the both phases and, at the same time, with suitable radioisotopes of the both components.

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