

## THE ROLE OF DIFFUSION IN ISOL TARGETS FOR THE PRODUCTION OF RADIOACTIVE ION BEAMS

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### ABSTRACT

On line isotope separation techniques (ISOL) for production of ion beams of short-lived radionuclides require fast separation of nuclear reaction products from irradiated target materials followed by a transfer into an ion source. As a first step in this transport chain the release of nuclear reaction products from refractory metals has been studied systematically and will be reviewed. High-energy protons (500 - 1000 MeV) produce a large number of radionuclides in irradiated materials via the nuclear reactions spallation, fission and fragmentation. Foils and powders of Re, W, Ta, Hf, Mo, Nb, Zr, Y, Ti and C were irradiated with protons (600 - 1000 MeV) at the Dubna synchrocyclotron, the CERN synchrocyclotron and at the CERN PS-booster to produce different nuclear reaction products.

The main topic of the paper is the determination of diffusion coefficients of the nuclear reaction products in the target matrix, data evaluation and a systematic interpretation of the data. The influence of the ionic radius of the diffusing species and the lattice type of the host material used as matrix or target on the diffusion will be evaluated from these systematics. Special attention was directed to the release of group I, II and III-elements. Arrhenius plots lead to activation energies of the diffusion process.

Results:

1. A strong radius determined diffusion behaviour was found:

$$\begin{array}{ccccccc} D_{\text{III B}} & > & D_{\text{II A}} & > & D_{\text{I A}} & > & D_{\text{VIII A}} \\ (D_{\text{Y}} & > & D_{\text{Sr}} & > & D_{\text{Rb}} & > & D_{\text{Kr}}) \end{array}$$

Rare earth elements diffuse as  $\text{Me}^{3+}$ -species.

2. Within the host elements of one period of the periodic table the diffusion of the trace elements changes in the following order:

$$D_{\text{III B}} > D_{\text{IV B}} \gg D_{\text{V B}} > D_{\text{VI B}}$$

3. In a given target trace elements of group I and II of a lower period diffuse faster than the corresponding elements of the higher period of the periodic table.

$$\begin{array}{ccccccc} D_{\text{2nd period}} & > & D_{\text{5th period}} & > & D_{\text{6th period}} \\ (D_{\text{Be}} & >> & D_{\text{Sr}} & > & D_{\text{Ba}}) \end{array}$$

The diffusion determined transport rate of nuclear reaction products in solid target materials is often satisfactory, and consequently several refractory metals are suited as targets for fast on-line separation of short lived nuclear reaction products. The delay times measured in on line mode at ISOLDE, however, are significantly shorter. An enhancement of the diffusion under radiation condition is considered, subject for further systematic studies. The results obtained in this systematic study may also be applied in the development of alternative separation technologies in medical radionuclide production.

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## 1. INTRODUCTION

Investigations of trace diffusion are not only important for our understanding of mass-transport phenomena in solids and liquids. Corresponding studies are often motivated from clear practical viewpoints as well. On line isotope separation techniques (ISOL) require a fast separation of the nuclear reaction products from the target material used and a corresponding fast transformation into a radioactive ion beam. This very complex process can be divided into the following separate transport steps:

- i. transport of the tracers to the surface of the target material
- ii. evaporation of the products from the surface
- iii. transport to the ion source
- iv. ionization and acceleration of the ions.

Under conditions where step ii can be neglected we are able to determine diffusion coefficients  $D$  from release measurements. For a homogeneous distribution of a radio tracer inside of a target matrix a special solution [1] of Fick's second law (1) [2] describes the relationship between the fractional release ( $F$ ) and the diffusion coefficient ( $D$ ), where  $d$  is the foil thickness and  $t$  the time (2):

$$\frac{dc}{dt} = D \frac{d^2c}{dx^2} \quad (1)$$

$$F [\%] = 100 - \frac{800}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left[ -\frac{(2n+1)^2 \pi^2 D t}{d^2} \right] \quad (2)$$

By introducing a diffusion number  $K = \pi^2 D t / d^2$  [6]  $F$  becomes only a function of  $K$ . This approach provides easy access to the diffusion coefficients  $D$  from release measurements.

Equation (2) is applicable only for foil-samples. Other geometric forms (powders, spheres or wires) require other solutions of the differential equation (1). Finally, the activation energy  $E_A$  of the diffusion process can be calculated after Arrhenius (3):

$$D = D_0 \exp \left( -\frac{E_A}{RT} \right) \quad (3)$$

From our systematic studies of the diffusion properties of a large number of tracers in different host materials we can learn more about diffusion mechanisms and can determine release half time (time required to release 50 % of the tracer) for conditions not available in off-line experiments. Related to the production of radioactive ion beams of very short-lived nuclei we are searching for conditions where the release half time is of the order of seconds or below. Thus, we are searching for conditions, where  $D$  becomes around  $10^{-7} \text{ cm}^2 \text{ s}^{-1}$  or larger, because under these conditions the release half time of nuclear reaction products would be of the order of 50, 10 and 0.5 sec for a target thickness of 100, 50 or 10  $\mu\text{m}$  respectively.

## 2. EXPERIMENTAL METHOD

Foils of refractory metals (Y, Ti, Zr, Hf, Th, Nb, Ta, Mo, W and Re) of different thickness (highest grade purity available, Goodfellow materials) and plates of dense graphite have been exposed to a proton beam of 600, 680 or later 1000 MeV of the Dubna or CERN synchrocyclotrons or the PS Booster at CERN. The irradiated samples are now doped homogeneously with a large number of radioisotopes generated via spallation reactions, fission or fragmentation. After a cooling period between 2 days and several months, samples of a few

mm<sup>2</sup> up to 100 mm<sup>2</sup> were annealed in vacuum ( $\leq 10^{-6}$  torr) under defined conditions. In order to getter oxygen traces the samples were usually covered with clean Ta foils. For further experimental details see [3]. The larger fraction of the presented experimental data have been obtained with a small electron bombardment furnace, that could heat a sample within one minute from room temperature up to 3000 °C [3]. The temperature region applied ranged from 1200 – 3000 °C and annealing time ranged from 1 – 100 minutes. The temperature was measured using a high temperature pyrometer and W(Re)/W thermocouples. Some samples were heated using a resistance heating facility inside a large vacuum recipient in a temperature range of 700 – 1700 °C. Finally, the third technology for annealing procedures made use of a standard vacuum facility and classical tube ovens (temperature region 600 – 1100 °C).

Standard gamma spectroscopy technique has been applied to determine the fraction of each individual released isotope simultaneously. Singles spectra were measured to very good statistics before and after annealing and analyzed by two different automatic analyzing codes as well as by manual inspection in particular to see that background subtractions were reasonable. Very good background spectra were recorded to be certain that no interference from background gamma-lines was present.

In order to avoid errors due to small changes in geometry between the measurements before and after annealing the spectra were normalized to each other by the observed activities of gamma lines belonging to long lived isotopes of the target matrix element, if applicable. For the determination of the Be release special efforts were required to analyze the gamma spectra. The samples, which usually contain a large excess of disturbing low energy gamma radiation, were measured through a combined filter of 3 mm Pb, 1 mm Cd and 0.5 mm Cu to stop the lower energy gamma ray. In this manner we could increase the activity load and were able to record the very few 478 keV photons emitted from the <sup>7</sup>Be traces present in the sample. These samples were measured overnight and analyzed after background subtraction. In other selected cases (release of Cs from Ta-matrix) radiochemical separation techniques were involved in order to increase the experimental resolution for the release of Cs [3].

### 3. FRACTIONAL RELEASE *F*

In Figs.1 we present some typical release curves recently measured. Fig.1a. illustrates the release of <sup>7</sup>Be from 1 mm thick graphite plates. For annealing time of 15 minutes the experimentally determined retention (*R*) of Be in the graphite samples is shown together with the corresponding fractional release (*F*-values). For 60 seconds annealing time only the *F*-values are shown determined in the same way experimentally. The *F*-values for 1 sec annealing time finally were calculated according to (2) based on the other experimental data.

In Fig.1b. we illustrate the fraction of released isotopes from 30 µm thick Y-foils after 20 minutes annealing time. We learn that Zn-traces are released faster than Be, which again is released significantly faster than Sr and Rb that show similar release properties. From Fig.1c we learn that from a Zr-matrix, one can release Y –traces at lower temperatures than Sr, while the Rb is released significantly only at temperatures close to the melting point (see also [8, 9]). Zn-traces diffuse slightly faster than Y and Be is even faster than Zn. Similar release curves were measured systematically for large number of radio-elements in 9 different host matrix materials [3, 8, 9, 11, 13, 14, 18]. We performed some measurements to access the release of radio-lanthanides from 50 µm Re-foils around 3000 K. The released fraction was so little that a quantitative interpretation was not possible for these conditions. The only conclusion we are allowed to do is to state, that the released fraction *F* for radio-lanthanides from a Re-matrix is significantly smaller compared to W-matrix under the same conditions. In order to obtain more precise data other experimental conditions (thinner foils ) are required.

#### 4. DIFFUSION COEFFICIENTS $D$

From the experimentally determined  $F$  values obtained under very different experimental parameters we determined diffusion coefficients  $D$  (equ.2) either by using the tables from Ziemen [4] or via the diffusion number  $K$  [6]. In order to illustrate how this approach works we present the Arrhenius plot for diffusion of  $Y$  in Mo matrix (Fig.2) [10]. In this graph we combined experimental data obtained for different foil thickness, different annealing times and an enlarged temperature region. A nice linearity is obtained independent of the different experimental parameters. This linearity was found throughout our systematic studies with only a few exceptions. The following figures (Fig's 3a-h) summarize the large data set in a compact way: Fig.3a. Arrhenius plot for the tracer diffusion in  $Y$  (this work), Fig.3b, 3c and 3d for the tracer diffusion in the group 4B matrix elements Ti, Zr and Hf [8, 9, 11, 18], Fig's. 3e and 3f for the tracer diffusion in the group 5B matrix elements Nb and Ta [13, 3] and finally, Fig's. 3g and 3h for the tracer diffusion in the Group 6B matrix elements Mo and W [9, 10, 14]. The exceptional non-linear Arrhenius plots are for the host elements of the Group 4B (Ti, Zr or Hf matrix, Fig's 3b-d, [8, 11, 12, 18]) and for the Se-release from Nb [13]. In the first case the typical phase transitions of the group 4B elements cause the non-linearity. The "normal" release of Se from the Nb-matrix [13] is probably disturbed by some chemical aspects.

From the Arrhenius equation we obtain the activation energy  $E_A$ , for the diffusion process and the pre-exponential factor  $D_0$  (Tab.1). These two values allow determining any diffusion coefficient in the temperature range indicated in the table.

#### 5. DISCUSSION

The large amount of diffusion data obtained from the different matrix systems and the applied standardized experimental conditions allow us to perform a very sensitive systematic data evaluation.

##### 5.1. Dependency of the diffusion on the matrix element:

By looking at the same trace element diffusing in different host lattices we observe a systematic trend showing a very strong decrease in diffusion speed as we go from group 4B via 5B to 6B hosts. This is shown in Fig. 4 where data from the diffusion of  $Y$  in several hosts is shown as well as data for some Lanthanides in Hf, Ta and W. When comparing the transport properties of the lanthanides (Ln) in Ta and W matrix, we learn that Ta should be the better target material for ISOLDE, because at a given high temperature the diffusion coefficients of radio-lanthanides in Ta are significantly higher (about factor 30) than in W (Fig's. 3f and 3h). The same systematic behavior can be seen for the other trace elements. The first general conclusion is: a trace element diffuses fastest in a matrix of the lower group of the periodic table:

$$D_{\text{IIIB}} > D_{\text{IVB}} \gg D_{\text{VIB}} > D_{\text{VIB}}$$

Following this conclusion, Hf and Zr should be the preferred target materials to achieve a fast release of the lanthanides and  $Y$  respectively. Corresponding solutions have been published [9, 12]. However, due to adsorption phenomena the release of the IIIB-group elements is significantly hampered in the applicable temperature region [15]. One can make an advantage out of that and gain selectivity by discriminating the "less volatile" elements as proposed in [9, 12] or to perform a fast radiochemical separation on the collector of on-line mass separators [16].

If we compare the diffusion of given trace elements in the matrix elements of period V and VI Mo and W (see Fig's. 3g and 3h) we learn that, for a given high temperature, Y-traces show a 30-fold higher diffusion coefficient in Mo ( $D_{Y \text{ in Mo}} > D_{Y \text{ in W}}$ ). The same systematic results can be seen for the matrix elements Nb and Ta (Fig's. 3e and 3f) and for Zr and Hf (Fig's 3c and 3d) respectively. Generally: a trace element diffuses faster in a matrix element of the lower period of the periodic table (Group IVB, VB and VIB) (Fig.4)

### 5.2. Ionic radius dependence of $D$

From all the preliminary data interpretation and discussion we learn that independent on the matrix element the diffusion behavior changes in the following order:  $D_Y > D_{Sr} > D_{Rb}$ . Consequently, one could conclude that the atomic radius of the diffusing species might be responsible for the differences. We compared the diffusion coefficients of the different diffusing trace elements in Ta-matrix with the atomic radii and, alternatively, with the ionic radii as well (see Fig.6 in [3]). No inhomogeneity in the  $\log D / \text{radius}$  plot was found in case of Eu and Yb. If Eu and Yb would diffuse as neutral or as  $Me^{2+}$ -specie, we should have seen a diffusion behavior similar to Ba. The only possible conclusion is, that the trace elements diffuse as species in 3+-, 2+-, 1+- and  $\pm 0$ -form for the IIIB-, IIA-, IA- and VIIIA elements respectively. In a Mössbauer study we learnt already, that in a metallic matrix (Fe) the  $^{151}\text{Eu}$  generated in a radioactive decay from implanted  $^{151}\text{Gd}$  becomes very fast stabilized in the  $\text{Eu}^{3+}$  state at room temperature [17]. In order to prove our empirical conclusion we did some additional efforts to measure the release properties of Kr in Zr [18] and Be in different materials (this paper). In Fig. 5a and Fig.5b. (Ta- and Zr- matrix respectively) we show 2 representative examples for the obtained radius dependency of the diffusion coefficient in a given matrix material.

These two figures 5a and b demonstrate the strongly decreasing diffusion speed from the 3+ elements (as for instance the lanthanides) over the earth alkaline (2+), alkaline (1+) to the neutral noble gases. In fact, it became very clear that not the vapour pressure of the diffusing element is determining the release properties; it is clearly the geometrical aspect. As a consequence of the above-developed philosophy traces of the group IVB elements (Zr or Hf) should diffuse faster than Y or the lanthanides due to the smaller ionic radius of the corresponding 4+-species. Actually, we can see a significant difference in the released Zr-fraction from Mo matrix depending on whether the sample was in contact with a Ta-surface (Zr.2) or not (Zr-1) (see Fig.5g [10]). One can conclude that, indeed, the diffusion of Zr in Mo (or Hf in W) is fast, but the release is hampered due to the very high adsorption enthalpies to the matrix surface [15].

### 5.3. Mass dependence of $D$

An inspection of Fig 5b also gives evidence of a mass dependence of the diffusion coefficient. The most characteristic example is Y, which is diffusing faster in Ta than the lanthanides that have equal or closely similar ionic radii.

## 6. THE DIFFUSION MECHANISM

There are different mechanisms at play for the diffusion in solids [6]. Diffusion in pure metals is mostly assumed to take place by the vacancy mechanism. An empirical relation [7] saying that the ratio between the melting temperature and the activation energy for diffusion in the matrix should be a constant has been shown to hold for a large number of diffusing species in many host materials.

The current understanding of a "normal" diffusion behavior comprises the following three aspects [19]:

- i The Arrhenius law must be able to describe the experimental data, whereby  $D_0$  and  $E_A$  are independent on the temperature. Deviations from the linear plot of  $\log D_0$  over  $1/T$  can be explained in terms of two or even more different diffusion mechanisms taking place simultaneously. Furthermore, anomalies in the Arrhenius plot can be induced by magnetic or crystallographic transformations, as seen for example in the diffusion behavior of lanthanides and Ba in Hf at the  $\alpha$ - $\beta$  phase transition point [11] (Fig.3d.).
- ii. The activation energy  $E_A$  is proportional to the melting temperature of the host metal  $T_M$  within an accuracy of  $\pm 20\%$ :  
$$E_A = 34 * T_M \quad [20] \text{ (given in [kcal/Mol])}.$$
- iii The pre-exponential factor  $D_0$  should be within the following values:  
$$0.05 < D_0 < 5 \text{ [cm}^2 \text{ s}^{-1} \text{]}$$

These three rules are generally accepted for self-diffusion in most of the metallic lattices. Deviations from these general rules are accounted as indications for non-reliable experimental results. For the tracer diffusion in metals very similar  $E_A$  values as for the self-diffusion were found as long as the tracer elements lie close to the host metal in the periodic table [21, 22].

From our systematic release studies we can clearly answer now the question about the advantages of refractory metals as target material over molten targets. In the introduction we formulated the requirement to find conditions for a given solid target metal, where the diffusion coefficient for the nuclear reaction product becomes  $D \geq 10^{-7} \text{ cm}^2 \text{ s}^{-1}$ , suitable to be used as on line target material. All investigated metals with the exception of W and Re, fulfill this preliminary requirement. (In case of  $25 \mu\text{m}$  Re-foil we could not measure significant fractions of radio-lanthanides even at the highest possible temperature 3200 K). As an example: according to presented experimental data the time for releasing 50 % of the radio-lanthanides out of  $50 \mu\text{m}$  Ta-foils is expected to be of the order of 30 sec at 2750 K. This is in absolute agreement with the Dubna experiences, where  $50 \mu\text{m}$  Ta-foils were used as target material in off line mode to produce short lived lanthanide isotopes in the framework of the JASNAPP Program (see for example [5, 23, 24]). A small roll of  $50 \mu\text{m}$  Ta foil (1 – 5 g) was irradiated with 680 MeV protons at the Dubna synchrocyclotron and then inserted into the surface ionization ion source [25] of the JASNAPP off line isotope separator. A few minutes (2-5) heating time was sufficient to release >95 % of the radio-lanthanides from such a Ta-foil target with the given foil thickness and a temperature up to 3000 K.

For comparison, the delay time for liquid metals as on line target has been measured to be in the region of 0.5 – 1 minute [26]. With very thin foils ( $\leq 10 \mu\text{m}$ ) one would be able to obtain a delay time in the region of seconds. However, the situation looks very different when we operate the target ion source system in on line mode, as it is the case at the ISOLDE facility at CERN [27]. The tantalum targets at ISOLDE are usually operated at lower temperature and the release properties measured in on line mode indicate significantly shorter release times compared to the off line measurements performed in Dubna. We believe that this effect is strongly related to the influence of the proton beam and is an additional argument for the "radiation enhanced diffusion" behavior. Further systematic studies are required in order to quantify the influence of different radiation modes and doses on the release properties of trace elements from a given matrix.

#### ACKNOWLEDGEMENT

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Fig's.1 Typical release curves [this work]

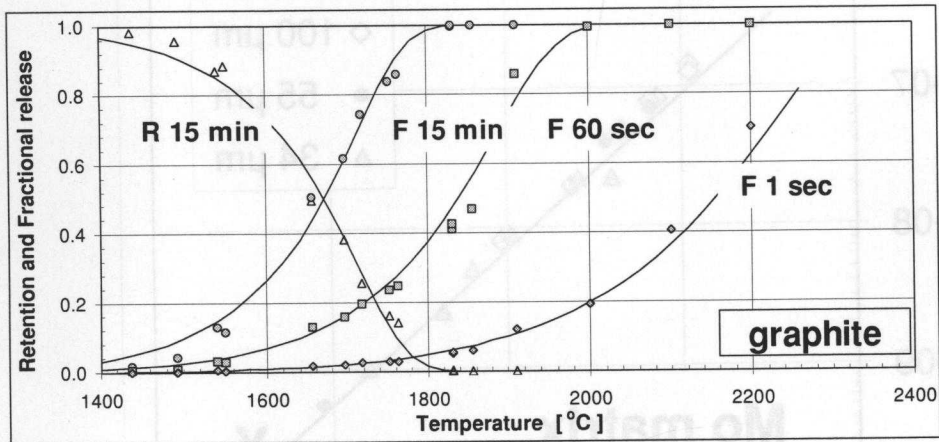


Fig.1a. Release of  $^7\text{Be}$  from 1 mm thick graphite plates (R = retained fraction, F = released fraction)

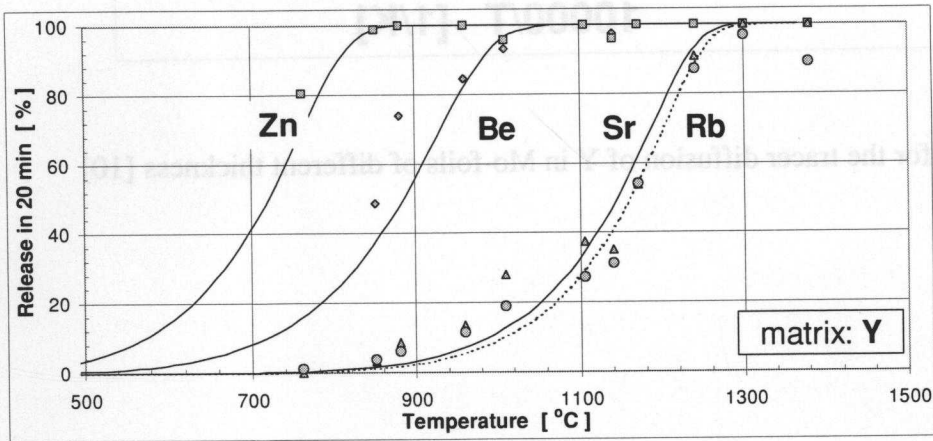


Fig.1b. Released fraction of nuclear reaction products from 30  $\mu\text{m}$  Y-foil for 20 min heating time

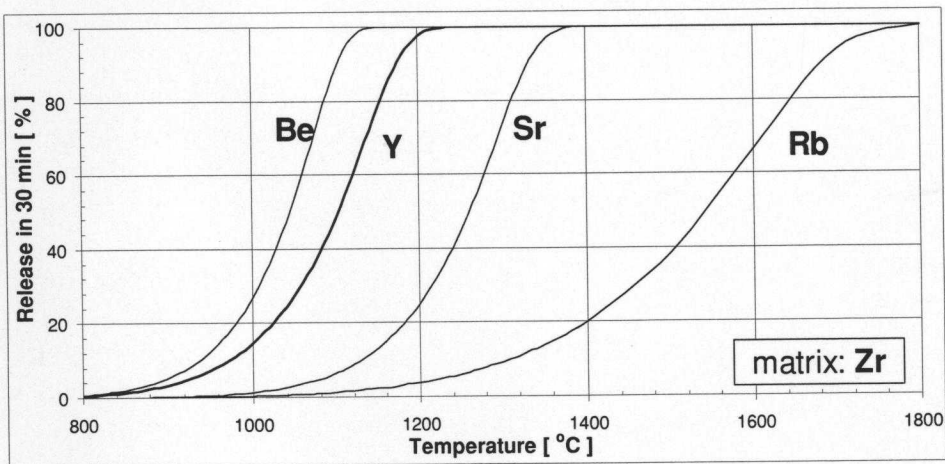


Fig.1c. Released fraction of nuclear reaction products from 100  $\mu\text{m}$  Zr-foil in 30 min heating time

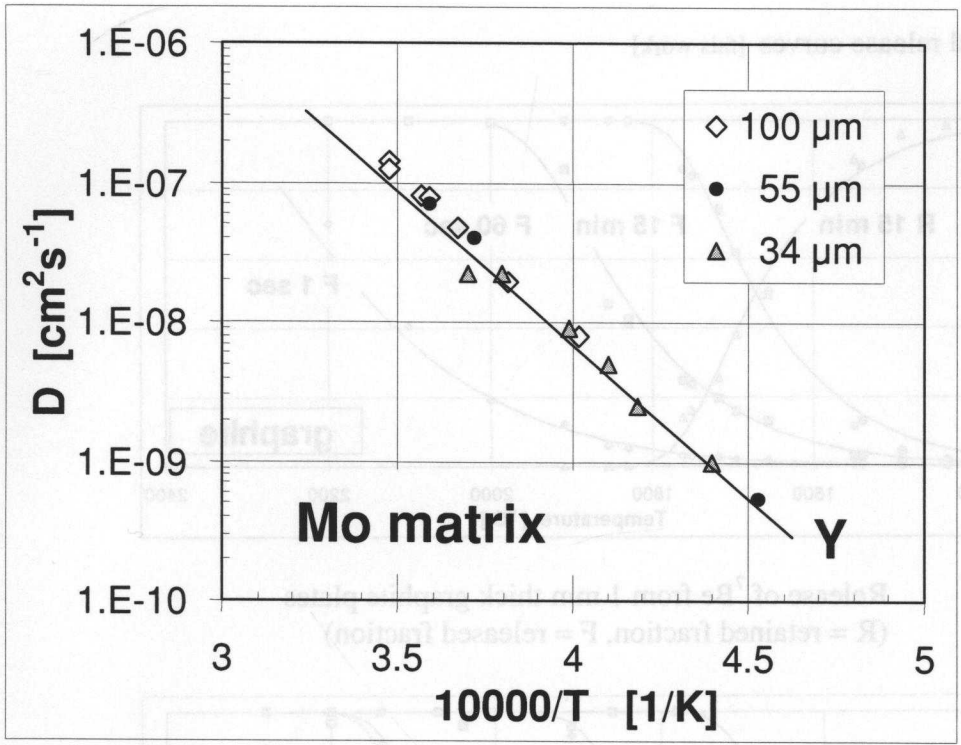
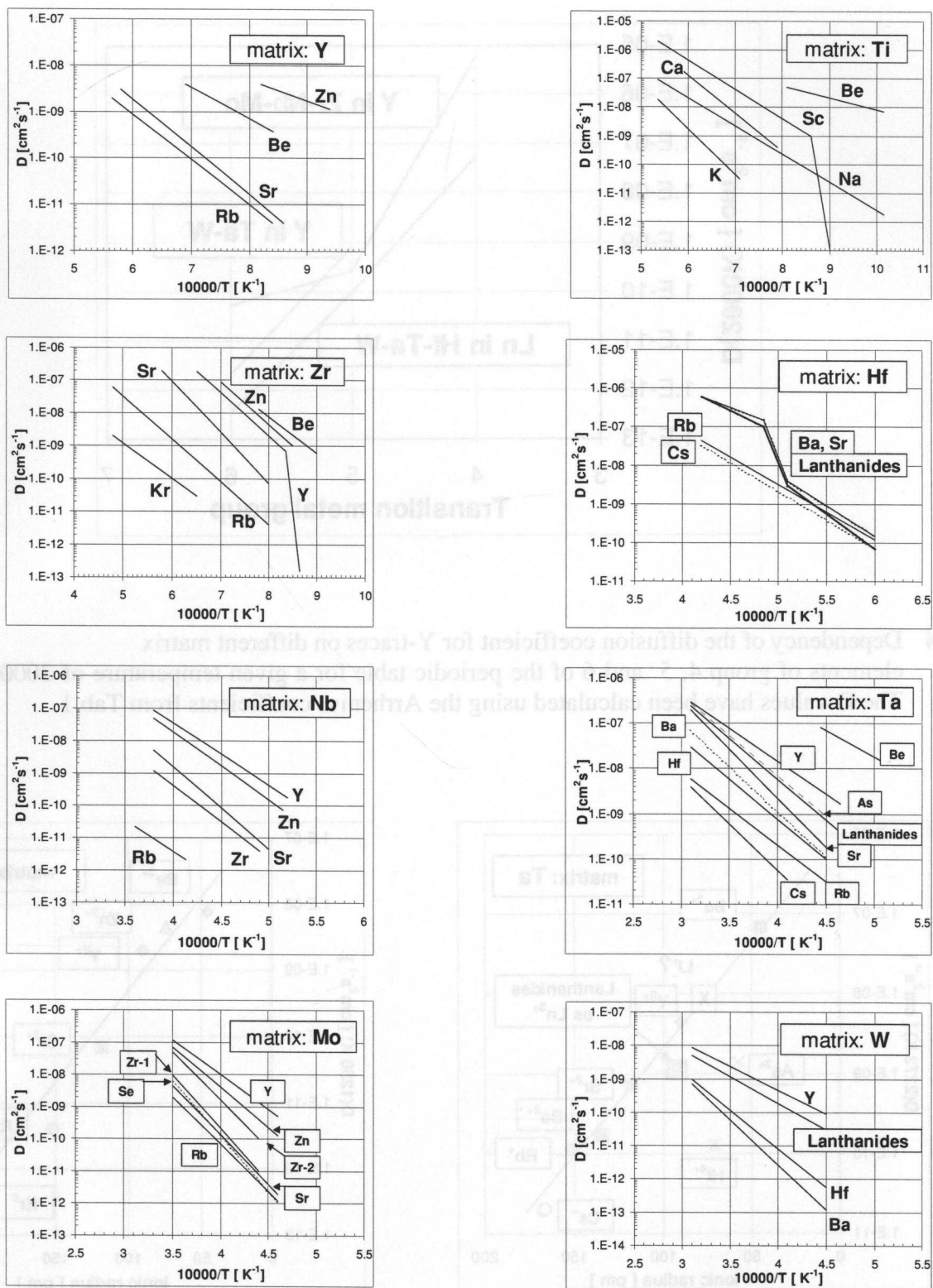
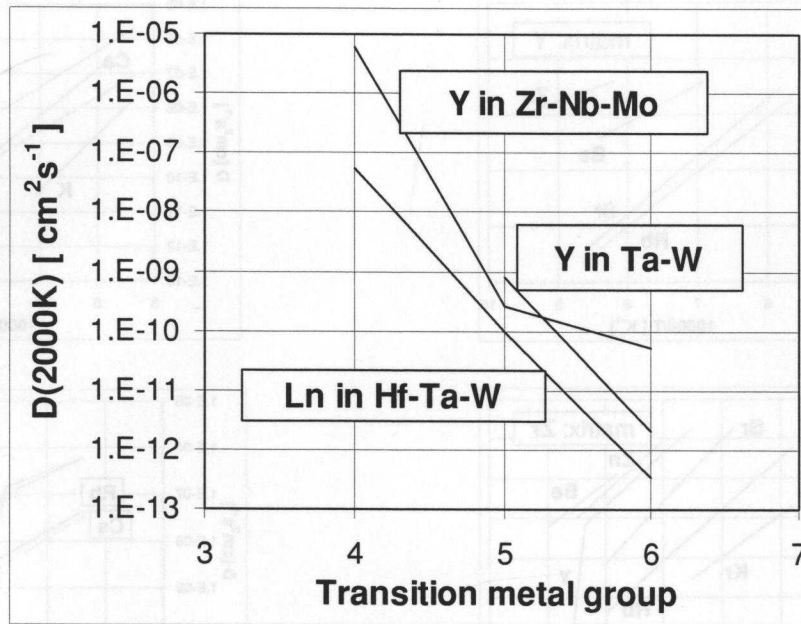


Fig.2 Arrhenius plot for the tracer diffusion of Y in Mo-foils of different thickness [10]

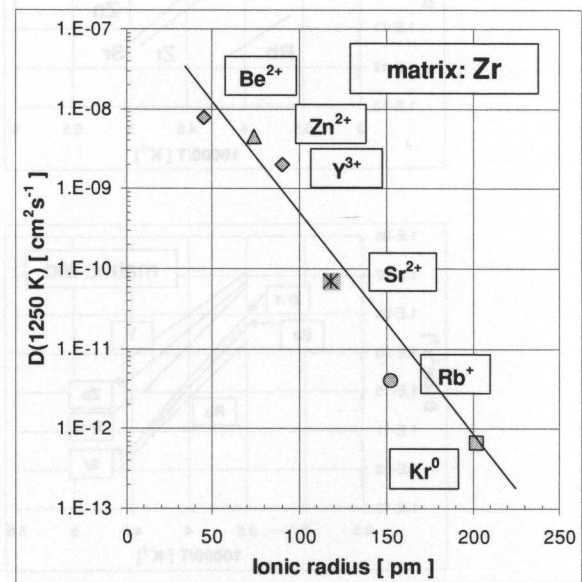
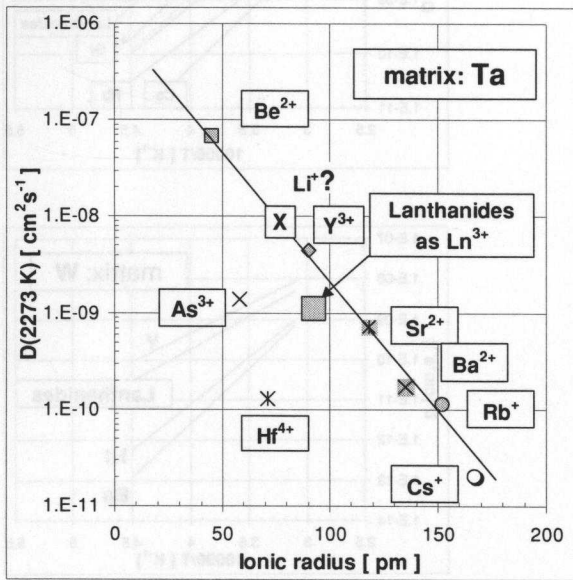




**Fig's.3** Arrhenius plots for the tracer diffusion of different nuclear reaction products in polycrystalline matrix of Y (Fig.3a.) and the refractory metals Ti (Fig.3b.), Zr (Fig.3c.), Hf (Fig.3d.), Nb (Fig.3e.), Ta (Fig.3f.), Mo (Fig.3g.) and W (Fig.3h.). The radio-tracers were generated via spallation reaction irradiating foils of the matrix element with high energy protons. After irradiation the radio-tracers are homogenously distributed throughout the matrix material. For more details see text and earlier published materials: tracer diffusion in Ti [8], Zr [8, 18], Hf [11], Nb [13], Ta [3], Mo [9, 10] and W [14]. (note: Zr-2 : the Mo-samples where wrapped with Ta-foil, Zr-1: the Mo-sample where annealed unprotected, for more details see ref. [9,10])



**Fig.4** Dependency of the diffusion coefficient for Y-traces on different matrix elements of group 4, 5 and 6 of the periodic table for a given temperature of 2000 K. The D-values have been calculated using the Arrhenius coefficients from Tab.1.



**Fig's.5** Dependency of the diffusion coefficient D on the corresponding radius of the diffusing species for different radio-tracers in polycrystalline Ta at 2000 °C ( Fig.5a.) and polycrystalline Zr at 977 °C (Fig.5b.). The corresponding D-values have been calculated from the Arrhenius coefficients in Tab.1. From this systematic one can extrapolate the diffusion coefficient for Li<sup>+</sup> in Ta matrix (Fig.5a.) to be  $D_{Li} = 10^{-8} \text{ cm}^2\text{s}^{-1}$ . This value has been confirmed by J.R.L.Bennett [28].

Host	Y		Ti		Zr		Hf		Nb		Ta		Mo		W		
T (K)	1000 - 1700		1200 - 1700		1200 - 2000		1700 - 2020		2000 - 2600		1700 - 3100		2200 - 2870		2400 - 3100		
Tracer	log Do	E <sub>A</sub> (kJ)	log Do	E <sub>A</sub> (kJ)	log Do	E <sub>A</sub> (kJ)	log Do	E <sub>A</sub> (kJ)	log Do	E <sub>A</sub> (kJ)	log Do	E <sub>A</sub> (kJ)	log Do	E <sub>A</sub> (kJ)	log Do	E <sub>A</sub> (kJ)	Tracer
Hf											-1.79	352			-1.81	439	Hf
Ln(**)							-0.1	274			-1.33	332			-1.54	418	Ln(**)
Lu							0.2	327			-1.63	321			-2.11	390	Lu
Yb							0.2	341			-1.65	318			-1.29	434	Yb
Tm							1.7	392			-1.24	336			-1.82	405	Tm
Tb							-0.8	301			-1.33	331					Tb
Gd							-0.6	306			-1.16	337			-0.71	466	Gd
Eu							-1.2	289			-0.96	347			2.12	390	Eu
Pm															-1.21	440	Pm
Ce															-1.54	426	Ce
Ba							-0.3	297			-0.67	395			1.03	620	Ba
Cs							0.5	358			-4.08	287					Cs
Zr									-0.32	436			1.75	502			Zr
Y					0.57	222			0.77	396	-2.8	241	0.97	431	-2.74	342	Y
Sr	-2.61	196			2.06	290	1.1	352	1.07	479	-1.36	338	1.88	565			Sr
Rb	-2.91	194			-2.14	231	0.2	322			-4.53	235	1.44	555			Rb
Kr					-2.79	227											Kr
Zn	-5.44	70			1.16	230			-0.77	411			-0.26	387			Zn
As											-0.9	346					As
Se													3.34	638			Se
Sc			0.23	207													Sc
Ca			1.74	270													Ca
K			-0.15	270													K
Na			-1.85	186													Na
Be	-4	120	-0.66	160	2.4	247					-1.78	234					Be
Ref.	this work		(8)		(8, 18)		(11)		(13)		(3)		(10)		(14)		

**Tab.1** Arrhenius coefficients according equation 3 for the tracer diffusion of nuclear reaction products (tracers) in the polycrystalline matrix of refractory host metals (host).

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