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THERMOCOUPLE COMPOSITION CHANGES DUE TO NEUTRON IRRADIATION

by

H. EHRINGER, C. MONGINI-TAMAGNINI and C. PONTI

1966

Directorate-General for Research and Training, Brussels

and

Joint Nuclear Research Center
Ispra Establishment - Italy

Scientific Information Processing Center — CETIS

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Summary

This paper mainly describes the methods employed to calculate thermocouple composition changes due to neutron irradiation in thermal reactors.

For each of the considered elements (Pt, Rh, Mo, Fe, Ru, Pd, Ni) a reaction chain has been constructed, and the corresponding set of differential equations has been solved by means of a FORTRAN program on an IBM 7090 computer.

Some interesting results concerning the reliability of the considered thermocouples are discussed.

Introduction

The in-pile testing of reactor materials and reactor operation require accurate temperature measurement in a neutron flux over a long period.Thermocouples provide the only practical means of temperature measurement at the precise location of the specimen while it is being irradiated.

The acauracy of the temperature measurement and the life-time of the thermocouple depend upon many factors, e.g. the temperature, time of exposure to high temperature, chemical reactions between thermocouple material and the specimen, diffusion,ect. When exposed to neutrons, thermocouples may undergo nuclear reactions, which change their composition and consequently their thermoelectric cheracteristics.

In most cases a thermocouple is considered reliable until it breaks and its output drops to nil. However, a long time before this poi t, its thermoelectric characteristics may have altered and this can only be detected by recalibration. In nuclear reactors, the magnitude of the composition change due to nuclear transmutation will not be negligible when the thermocouple is used in a high flux reactor for several days or when it is exposed to a less intense neutron field for a very long time.

From these facts it can be concluded that the life time of a thermo couple depends not so much on this material as on its operating conditions, which vary from one application to another.

Some measurements of the in-pile changes in EMF characteristics of Chromel/Alumel and Platinum/Platinum-Rhodium thermocouples were carried out by M.J.Kelly[1][2] and by C.W.Ross[3]. They showed

Manuscript received on August 16, 1966.

that the net output of the Chromel-Alumel-thermocouples used in their experiments was not correlated with exposure to neutrons ($< 4,10^{20}$ n.v.t.) and the observed errors remained within the allowed tolerances. In the case of Pt/Pt-Rh thermocouples, however, the observed deviation was correlated with the neutron exposure and the error above 10^{20} nvt was not longer negligeable. The changes in the thermoelectric properties of W/W-26 Re Thermocouples are even greater; the observed deviation for initial calibration reached values up to 80°C at temperatures of about 1000°C after 2.5. x 10^{20} nvt[4].

The radiation-induced changes in the composition of Chromel/Alumel, Pt/Pt 10% Rh and W/W 26% Re thermocouples were calculated by W.E.Browning Jr. and C.E. Miller Jr.[5] These calculations confirm the sensitivity of the Pt/Pt Rh thermocouple.

From the results of all these studies it can be concluded that for the temperature range 0-1000°C, Chromel-Alumel thermocouples can be used in nuclear reactors up tc neutron exposures of at least 5 x 10^{20}nvt. For the temperature range above 1000°C the commercially available thermocouple Pt/PtRh and W/W 25% Re are no more suitable if the neutron exposure exceeds about 10^{20} nvt.

Consequently there exists a need for new thermocuple alloys which are less sensitive to neutrons. In this study, the transmutation effect of various binary platinum alloys was calculated for thermal neutron fluxes of 0,5, 1,2 and 4 x 10^{14} n/cm^2 S. up to thermal neutron doses of 10^{23} nvt. In the choice of the components and alloy composition, mechanical, physical, thermoelectrical,chemical, metallurgical and nuclear characteristics were taken into account.

For temperatures above 1000°C only noble or refractory metals can be considered as thermocouple materials. Other metals can be used as alloying components if their vapour pressure is low at the melting point of the alloy.

In this study platinum alloys were considered, the aim being to obtain lower transmuation rate than Pt/Pt 10% Rh under reactor conditions. Among the platinum metals, only Ru, Pd, Os and Pt have relatively small neutron cross-sections, but osmium can be ruled out owing to its hexagonal crystal structure and its unfavorable metallurgical properties. The melting point of palladium is fairly low (1554°C) and both its vapor pressure and its diffusion coefficient must be taken into account at high operating temperatures. With platinum it forms an uninterrupted series of solid solutions. The thermoelectric behaviour of Pt-Pd alloys is described by R.F.Vines and E.M.Wise[6].

Ruthenium melts at 2250°C and its solid solubility in platinum exceeds 66 wt%. The thermoelectric emf of Pt-Ru alloys is higher than the corresponding values for Pt-Pd alloys.

Iron and nickel also have small neutron cross-sections and can be used as alloying components with platinum. Metallurgical and physical studies are being carried out in order to examine their suitability as thermocouple material.

Platinum-molybdenum thermocouples are commercially available and can be used in unclear reactors owing to the low neutron cross-section of molybdenum. The use of this thermocouple at temperatures above 1300°C is limited because of the chemical properties of

the molybdenum. Little is as yet known about its in-pile transmutation effect.

Experience shows that impurities in concentration higher than 0.1% can change the thermoelectric characteristics considerably and the resulting effect is a function of the nature of the impurities.

In order to predict the change in the thermoelectric curve of a thermocouple as a function of the neutron exposure it is necessary to prepare alloys containing the calculated elements which are formed by transmutation of the virginal thermocouple alloys, their concentration being known from transmutation calculations.

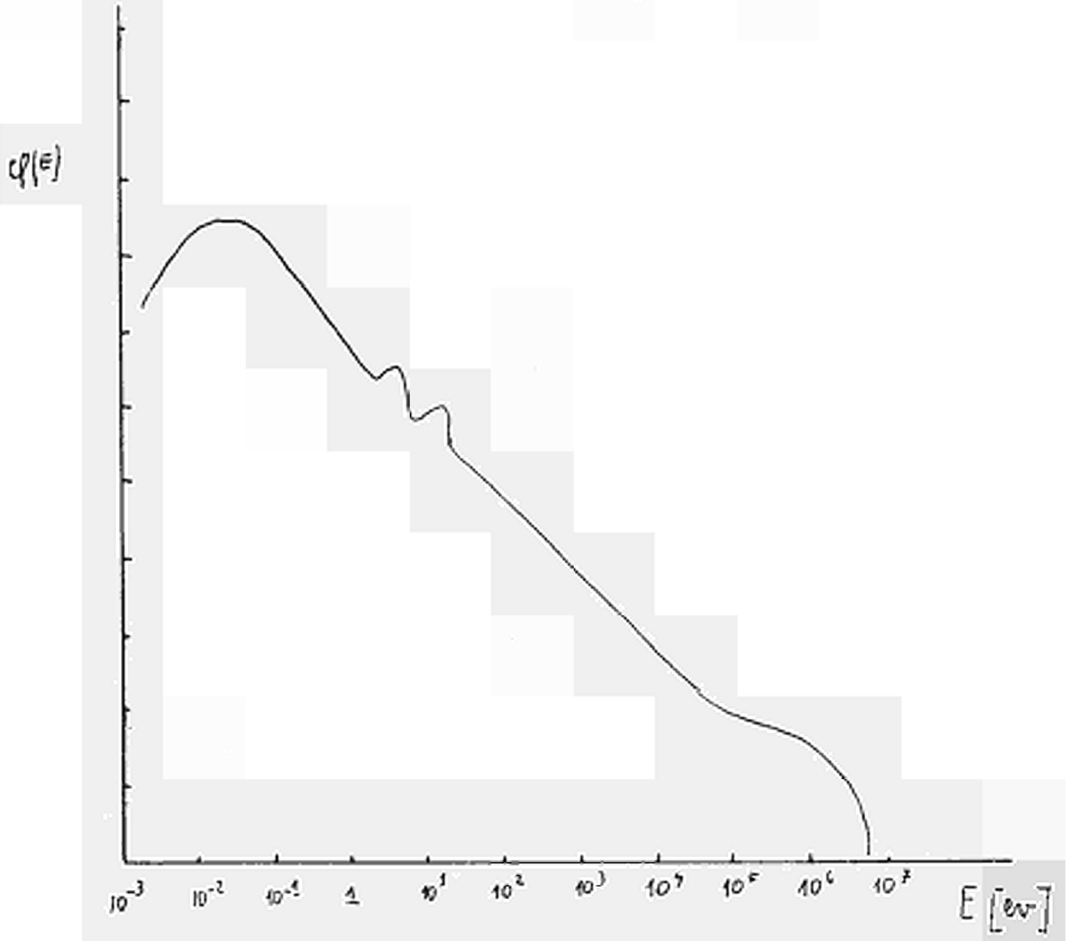
In case of difficulties in the alloy preparation, in-pile irradiation experiments and post-irradiation recalibration are necessary for estimating irradiation-induced changes in the thermoelectric characteristics.

Calculation of thermocouples composition as a function of irradiation

1. The one-group thermal model

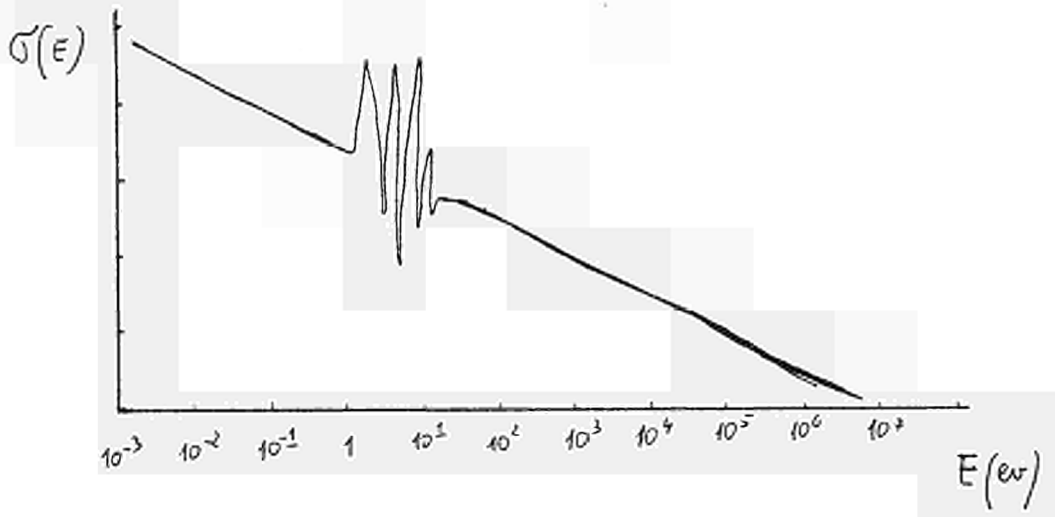
When studying the effect of irradiation on elements or compounds the entire range of neutron energies ought to be considered. As known in a nuclear reactor the neutron energies range from approximately 10^{-3} to 10^{+7} electron volts, and the classification in thermal, epithermal, fast reactors, corresponds in fact to a prevalent neutron density respectively in the thermal, epithermal or fast zone.

In the case of thermal or nearly thermal reactors, like the ones here considered, namely the BR2 at Mol and the HFR at Petten, the qualitative behaviour of the neutron spectrum is the following:



As for the capture cross sections of the isotopes appearing in the time dependent composition of the considered thermocouples, they show a qualitative behaviour of this type. The resonances

are not present for all the isotopes, but for some of them[7] (Mo, Ru, Rh, Pd, Pt, Ag, Au, Hg) they are very strong.



A procedure which can be used for the calculation of irradiation effect on composition, when very precise results are wanted is the following: first of all the neutron spectrum in the considered reactor is calculated, by means of a nuclear code like GGC[8]; then group cross sections are obtained in some (e.g.3 or 4) broad energy groups, by averaging on the spectrum. Finally the differential equations describing the composition changes vs. time for each isotope in each broad group are written and solved.

Though such a procedure gives very good results (as far as the nuclear data are to be trusted) it is rather cumbersome, and in the case of thermal reactors generally other models are used, much more straightforward.

The most popular, the one group Westcott model[9], assumes the neutrons to be all of the same energy, corresponding to the thermal peak.

As for the cross sections the Westcott expression is the following:

$$\hat{\sigma} = \sigma_0(g+rs)$$

where σ_0 is the cross section corresponding to 0.025 electron volt, g and s give a measure of the departure from the 1/v law respectively in the thermal and epithermal zone, while r is related to the ratio between the thermal and epithermal fluxes (r ranges between 0 (all thermal fluxes) and ~1 (all epithermal fluxes)). g and s, which depend on temperature, are tabulated for the different isotopes.

In this paper, which treats in fact a first approach to the study of the behaviour of thermocouples under irradiation, some further hypothesis have been done: more precisely g has been set = 1 (tha is 1/v behaviour for the thermal cross sections has been assumed) and the product rs has been neglected in respect to 1 (that is we have suppoed a small epithermal flux and hence we have neglected the resonances in epithermal region).

The first hypothesis seems to be quite valid. As for the second one, it is in our intention to remove it in future; however we are convinced that also with the present hypothesis the results we have got permit us to have a good insight into the behaviour of thermocouples under irradiation in the considered reactors.

2. Nuclear reaction chains

As already stated the thermocouples considered in this paper have the following compositions (in weights %);

1) Pt 100%

2) Pt 90% + Rh 10%

3) Pt 99,9% + Mo 0.1%

4) Pt 99% + Mo 1%

5) Pt 95% + Mo 5%

6) Pt 99.97% + Fe 0.03%

7) Pt 99% + Fe 1%

8) Pt 99.9% + Ru 0.1%

9) Pt 99% + Ru 1%

10) Pt 98% + Ru 2%

11) Pt 97% + Ru 3%

12) Pt 96% + Ru 4%

13) Pt 99% + Pd 1%

14) Pt 80% + Pd 20%

15) Pt 73% + Pd 27%

16) Pt 95,5% + Ni 4,5%

Let us consider the nuclear reaction chains of the elements: Pt, Rh, Mo, Fe, Ru, Pd, Ni appearing in the above list. For each of the elements a chain has been prepared (see annexed tables) in which the values of σ_0 and of the half lives are reported. Nearly all the values for the cross sections and half lives, have been picked up from BNL 325[7] and ANL 5800[10], or the Handbook of Nuclear Physics.

However, in the case of unusual information sources, we have indicated the corresponding paper.

In certain particular cases the cross sections were not known, or

were given with an error of the order of magnitude of the cross section itself.

In such cases we have tried to vary the cross sections in reasonable intervals and fortunately we have found that the results obtained for the element concentrations during time are not significately affected by the variations.

The same is not true for the isotopic concentrations, of course, but when studying thermocouples changes due to irradiation only the element concentrations are important.
We will list on the following the uncertainties encountered.

Pt chain

$\sigma(Pt^{193})$ is not known, and we have hence finally assumed a zero value for $\sigma(Pt^{192})$

$\sigma(Mo^{193})$ is not known, and as for the case of Pt we have finally assumed $\sigma(Mo^{192})=0$.

$\sigma(Mo^{198})$: two data are given. In such case the difference obtained on elements concentration are somewhat more important, and in certain cases both the resulting set of curves have been reported (see fig.Nr.3 and Nr.16). Elsewhere only the value $\sigma=0.18$ has been retained.

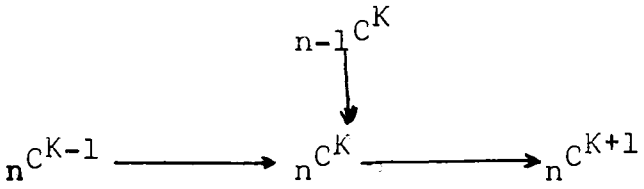
Ni chain

- A variation of $\sigma(Ni^{59})$ between 0 and 10^4 does not alter the final results of element's compositions.
- $\sigma(Ni^{61})$ is 2b with an error of ∓ 1b. The essays with the values 1, 3 gave the same results.
- $\sigma(Ni^{63})$ is not well known, but the results for elements do not depend on it.

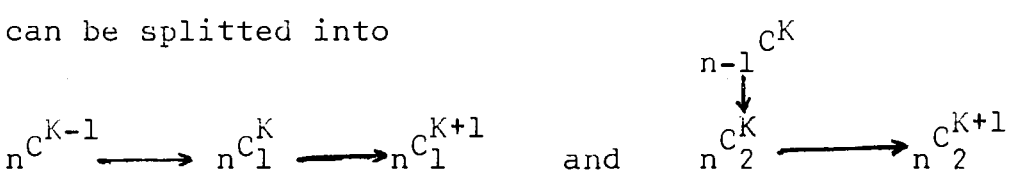
3. Methodological aspect and discussion on the results

Let's consider first the reaction chains originated by the single elements Pt, Rh, Mo, Fe, Ru, Pd, Ni.

First of all whenever an isotope is born from two different processes, for instance a decay and a capture, the chain can be splitted into two separate chains, and the final results will be then summed up. That is the chain



can be splitted into



where $_nC^K = {}_nC_1^K + {}_nC_2^K$ $\qquad$ $_nC^{K+1} = {}_nC_1^{K+1} + {}_nC_2^{K+1}$

In such a way the general balance equation to be solved for each isotope of the chains is :

$$\frac{d\,{}_nC^K}{dt} = \begin{pmatrix} {}_n\sigma^{K-1}\varphi \cdot {}_nC^{K-1} \\ \text{or} \\ {}_{n-1}\lambda^K \, {}_{n-1}C^K \end{pmatrix} - ({}_n\sigma^K\varphi + {}_n\lambda^K)\,{}_nC^K$$

Whenever ${}_n\sigma^K\varphi + {}_n\lambda^K$ is very big as compared to ${}_n\sigma^{K-1}\varphi$ or ${}_{n-1}\lambda^K$, we can suppose that the equilibrium state is reached

very soon and we can set

$$ {}_nC^K = \frac{\left({}_n\sigma^{K-1}\,\varphi\,{}_nC^{K-1} \right) \quad \text{or} \quad \left({}_{n-1}\lambda^K\,{}_{n-1}C^K \right)}{({}_n\sigma^K\,\varphi + {}_n\lambda^K)} $$

In such a case the isotopes derived by ${}_nC^K$ that is ${}_nC^{K+1}$ and ${}_{n+1}C^K$, can be considered as derived directly from ${}_nC^{K-1}$ or ${}_{n-1}C^K$.

Moreover, whenever ${}_n\lambda^K$ is small as compared to ${}_n\sigma^K\varphi$, and the isotope is derived from capture, the equation can be written:

$$ \frac{d\,{}_nC^K}{d\theta} = {}_n\sigma^{K-1} \cdot {}_nC^{K-1} - {}_n\sigma^K \cdot {}_nC^K \text{, where } \theta = t.\varphi $$

Numerical experiences have shown that, for the fluxes here considered ($0.5.10^{14}$, 1.10^{14}, 2.10^{14}, 4.10^{14}), and when interested in the long-range thermocouple irradiation (order of months, or years) then the nuclear chains reactions can be assumed to be the ones given by the dotted lines in the annexed tables. We want to note again that this is true for element concentrations, and not for the isotopic ones.

As a consequence one can see that only for the elements Ni and Fe, it is important to consider as independent variable the time; for the other elements θ can be used as well.

The composition changes for Pt - Rh - Mo - Fe - Ru - Pd - Ni as obtained by means of numerical calculations (see next paragraph) a shown in fig.1 to 13 respectively.

Note that this compositions are in atom percent. However they can be considered, to a good approximation, also compositions in weigh percent since differences only of two or maximum three units over hundred are found in the weights of the elements considered.

As for the thermocouples to be studied, apart of the case of pure Platinum we have just to weight and sum together the results for t single elements in order to have the results for the given compou The results obtained are collected in fig. from 14 to 41.

4. Numerical method.

As we have said before, the nuclear reaction chains arising from a particular element, can be splitted into a number of individual chains in which each element is born from the preceding one and goes into the next one. The set of equations corresponding to a individual chain can be written in its general form as follows:

$$\frac{dW_1}{dx} = - B_1 W_1$$

$$\frac{dW_2}{dx} = A_1 W_1 - B_2 W_2$$

$$\frac{dW_3}{dx} = A_2 W_2 - B_3 W_3$$

.....

.....

with given initial conditions $W_i(0) = W_{io}$.

Two different numerical approaches have been utilized for the solution of the above system. In both the cases a program (in Fortran language) has been prepared for the IBM 7090 of CETIS, C.C.R.EURATOM, Ispra.

The first approach is to solve the above system of differential equations by means of a double precision integration routine based on Runge-Kutta method.

The second one consists in solving analytically the above system; in fact one can easily verify that:

$$W_{J+1} = \sum_{i=1}^{J+1} a_{J+1,i}\, e^{-B_i t}$$

where

$$a_{J+1,J+1} = W_{J+1}(o) - \sum_{i=1}^{J} a_{J+1,i}$$

$$a_{J+1,i}\,(i=1,2\ldots J) = \frac{A_J}{B_{J+1}-B_i}\, a_{Ji}$$

The W terms are then calculated by means of the computer. It is evident that such recursive formulae can be applied only when $B_i \neq B_J$ for $i \neq J$; even when B_i and B_J are of the same order some numerical indeterminations can easily arise in the results. Fortunately this was not our case.

For all the chains arising from the considered elements the two mentioned schemes were utilized, giving practically the same results, which are then to be regarded as very satisfactory.

Preliminary conclusions

Pure platinum forms mercury and gold by transmutation, the concentration of which cannot be ignored at integrated neutron fluxes above 10^{21}n/cm^2. The Pt 10% Rh wire, however, forms palladium in concentrations exceeding 0,2 and about 1% after 10^{20} and 10^{21} n/cm^2 respectively. Consequently the thermocouple Pt/Pt 10% Rh cannot be regarded as reliable after neutron exposures exceeding 10^{20}n/cm^2.

The transmutation effects of iron-,cobalt-,palladium-,ruthenium- or tungsten-containing platinum- base thermocouples can probably be ignored up to an integrated neutron flux of 10^{21}n/cm^2, since the concentration of the formed elements remains at values of less than 0.71%. In fact their composition change is mainly determined by the platinum transmutation.

x
x x

Acknowledgements

We would like to thank Dr.ROCCA (CETIS) for the help given in finding out the cross sections for many isotopes from the literature.

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(7) BNL-325 .

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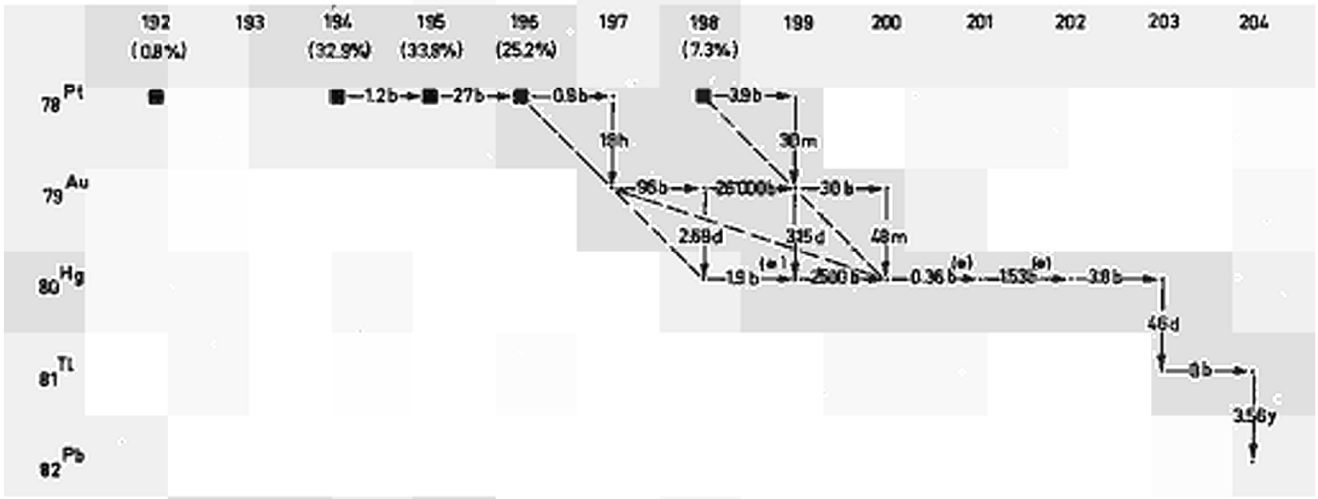
(9) C.H.WESTCOTT, W.H.WALKER, I.K.ALEXANDER, Geneva Conference 1958.

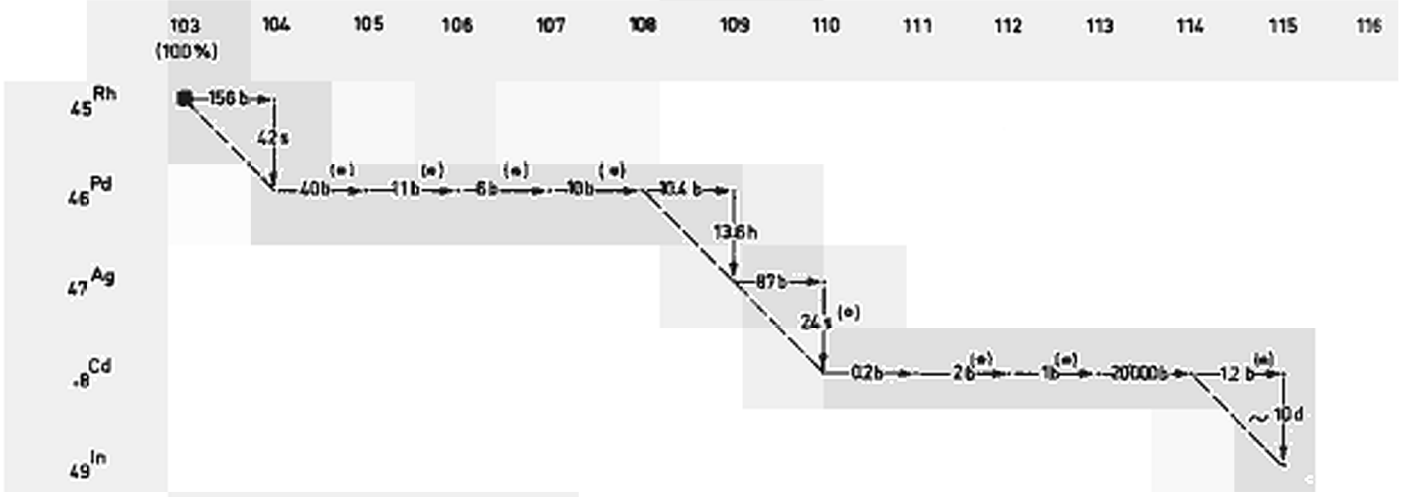
(10) ANL-5800.

Pt CHAIN



(*) From private communication

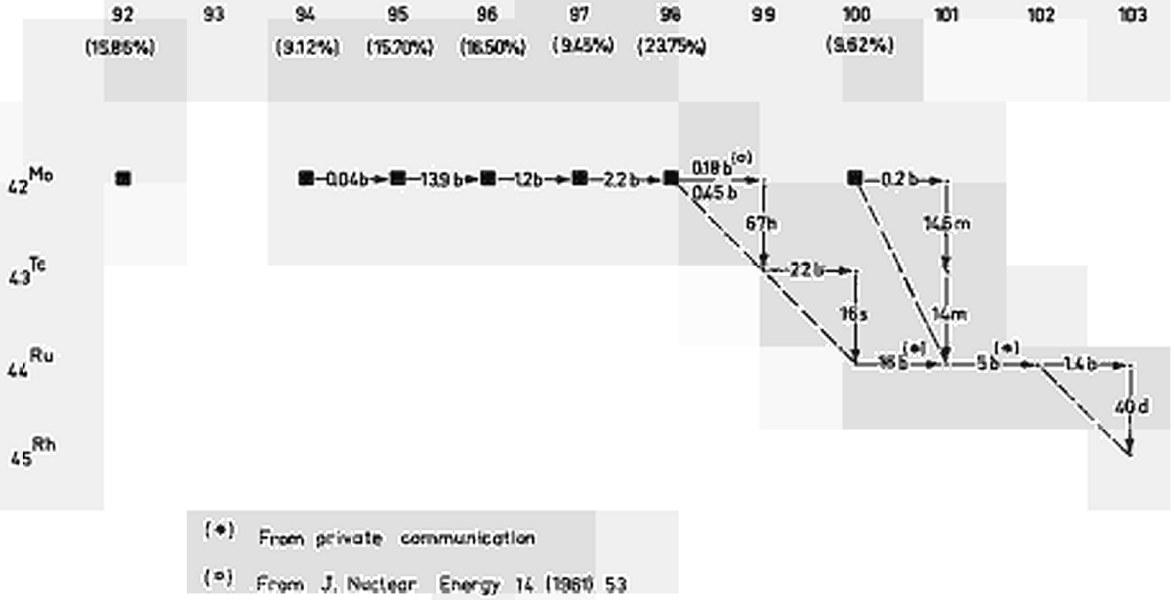
Rh CHAIN



(*) From private communication

(°) From Phys. Rev. 129 (1963) - 769

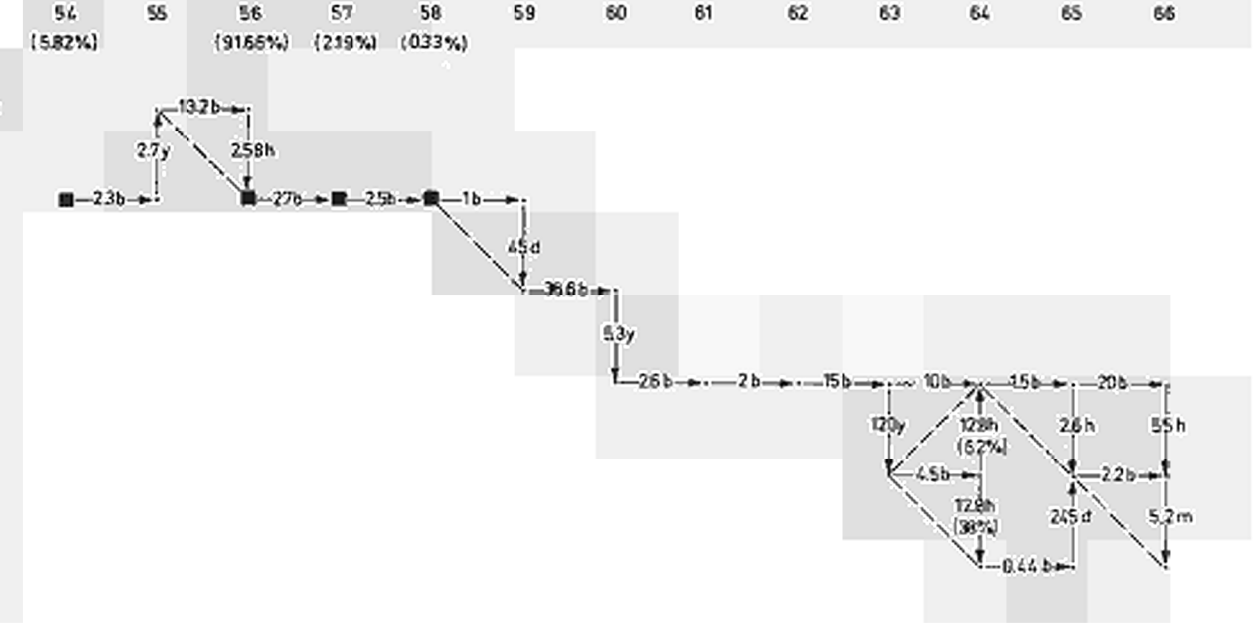
Mo CHAIN



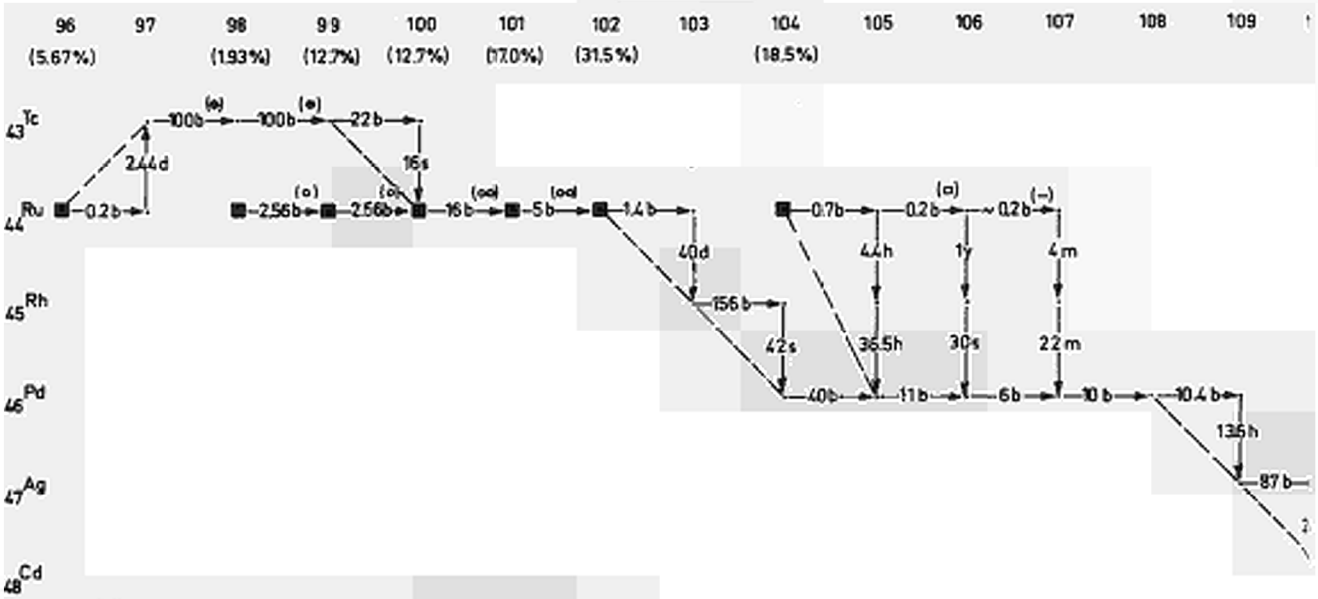
(*) From private communication

(o) From J. Nuclear Energy 14 (1961) 53

Fe CHAIN



Ru CHAIN



(e) The cross section of natural Tc has been assumed

(o) The cross section of natural Ru has been assumed

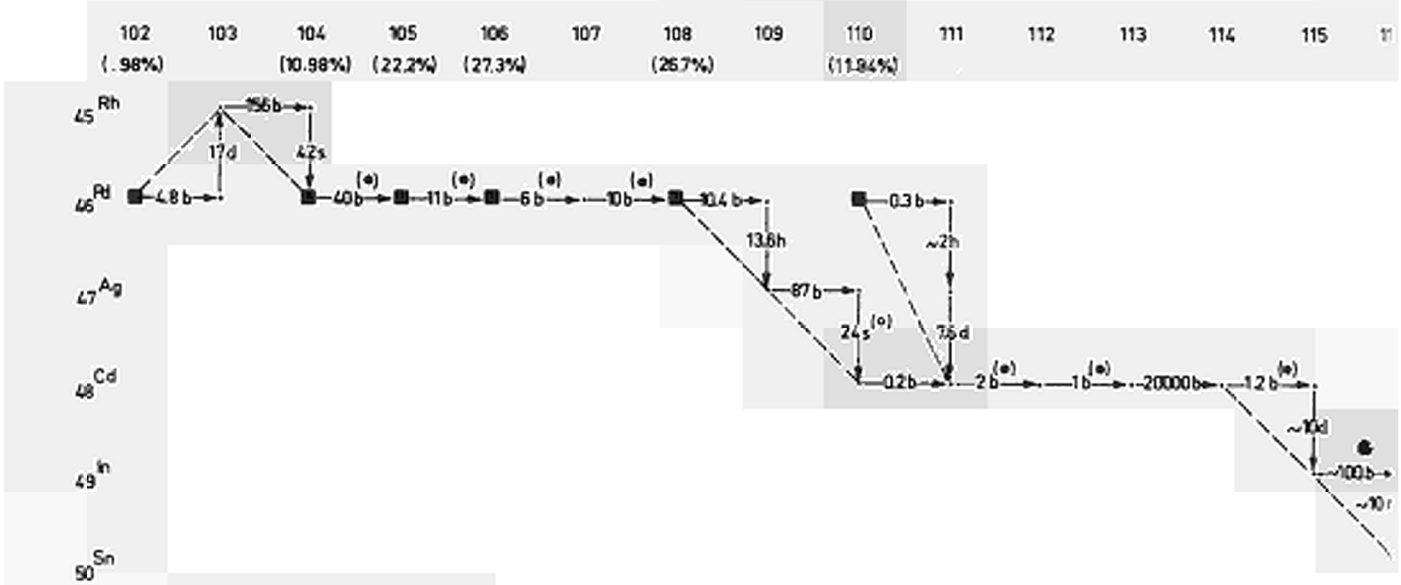
(oo) From private communication

(a) From Nuovo Cimento (10), 17, 887

(-) The cross section of Ru^{105} has been assumed

Fig. 1

Pd CHAIN



(e) From private communication

(o) From Phys. Rev. 129 (1963) 769

Ni CHAIN

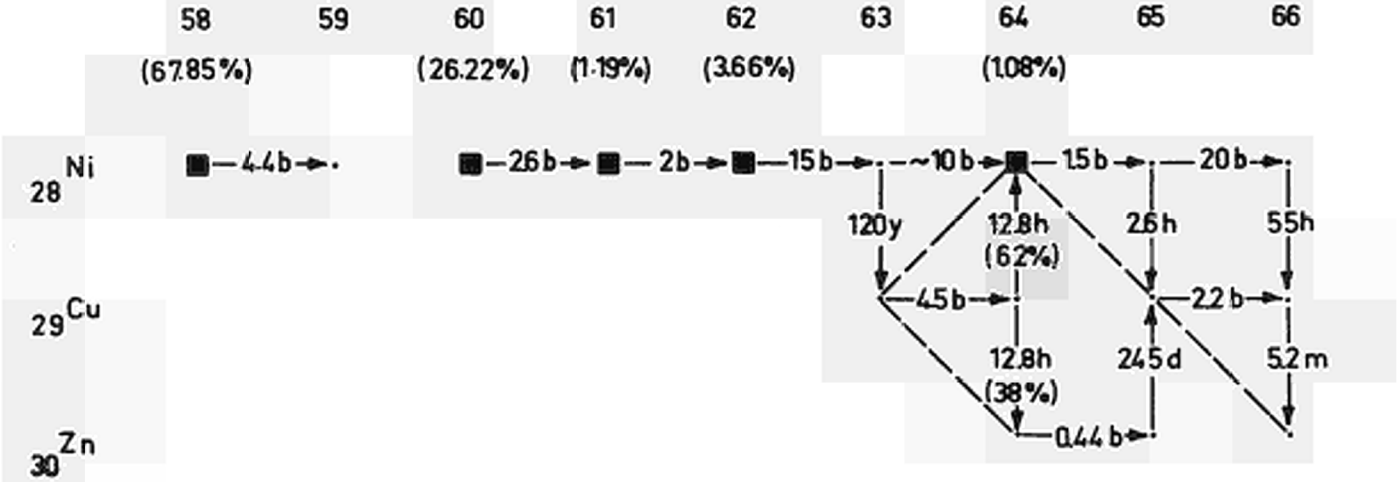
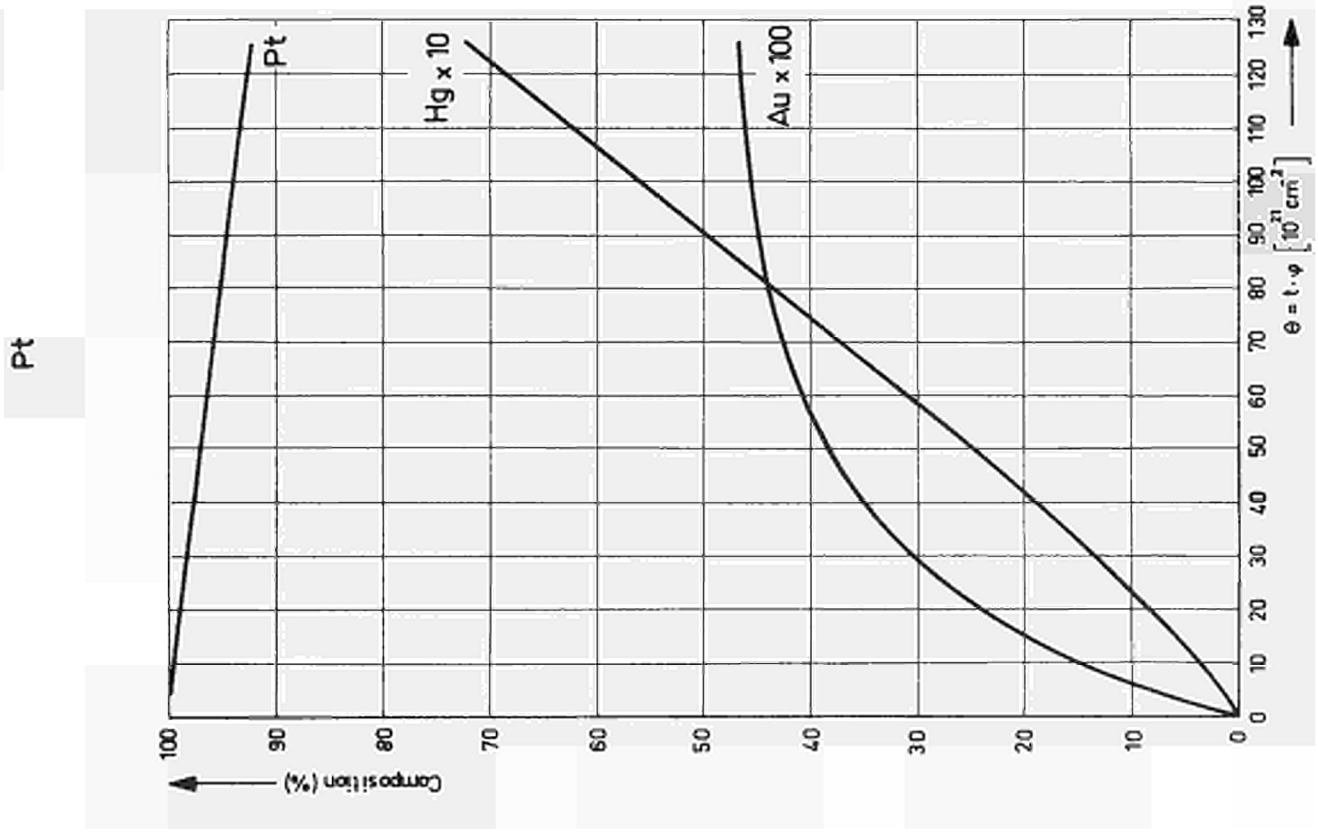


Fig. 1



Pt

Fig. 2

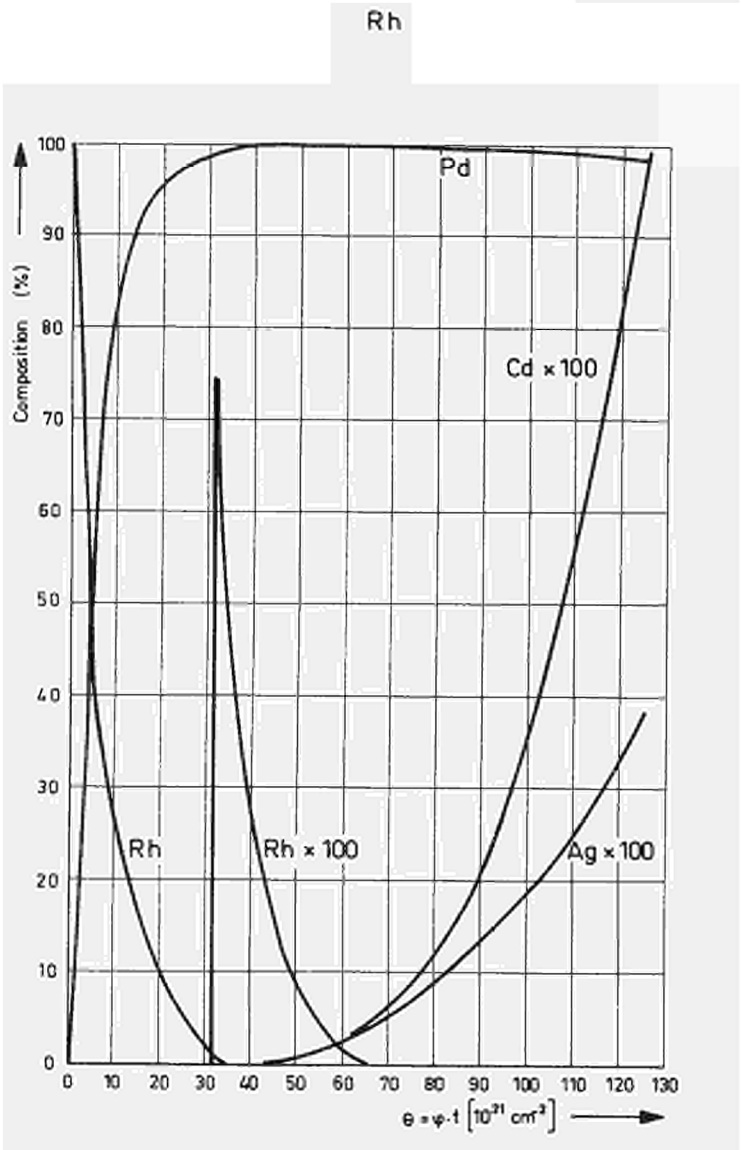

Rh
Composition (%)
Pd
Cd × 100
Rh
Rh × 100
Ag × 100
θ = φ·t [10²¹ cm⁻²]

Fig. 3

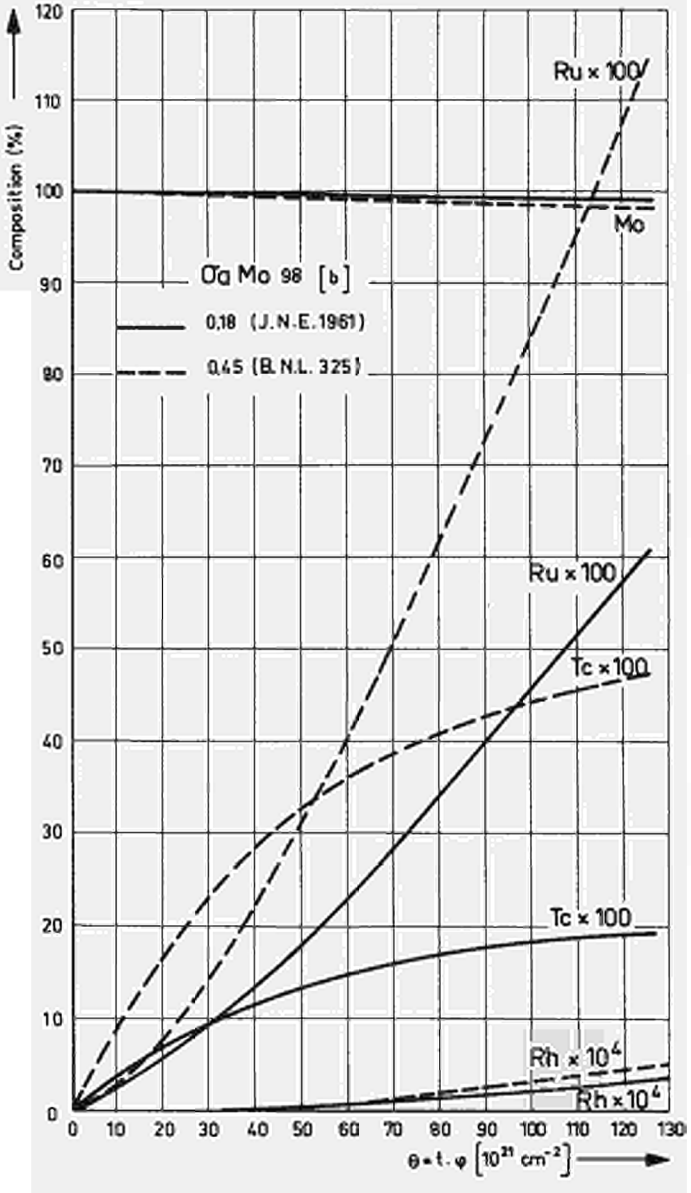

Mo
Composition (%)
Ru × 100
Mo
Ca Mo 98 [b]
0,18 (J.N.E. 1961)
0,45 (B.N.L. 325)
Ru × 100
Tc × 100
Tc × 100
Rh × 10⁴
Rh × 10⁴
θ = t · φ [10²¹ cm⁻²]

Fig. 5

Iron

Thermal neutron
flux $1. \times 10^{14}$ n cm^{-2} sec^{-1}

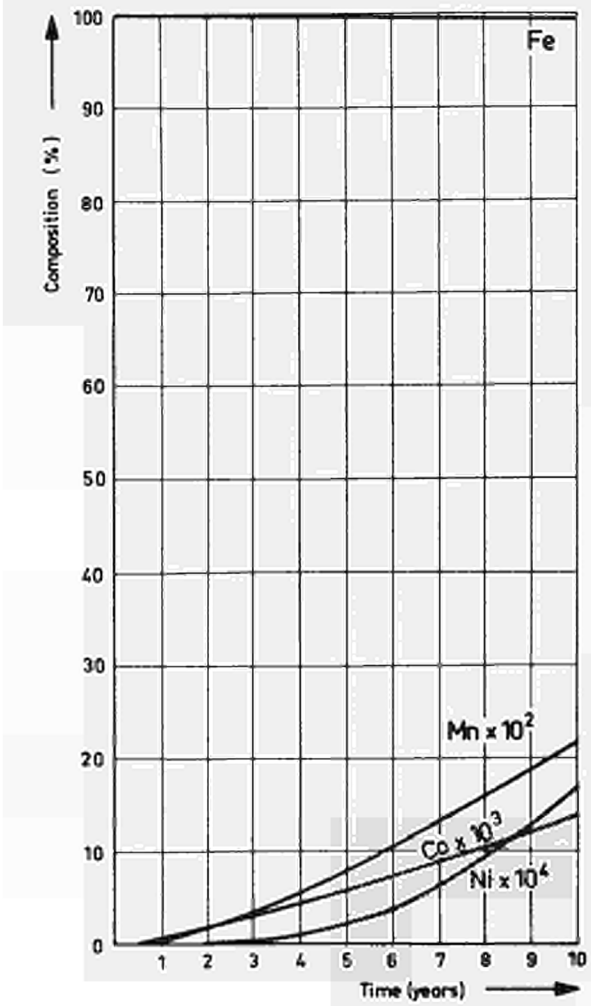


Fig. 4

Iron

Thermal neutron
flux $.5 \times 10^{14}$ n cm^{-2} sec^{-1}

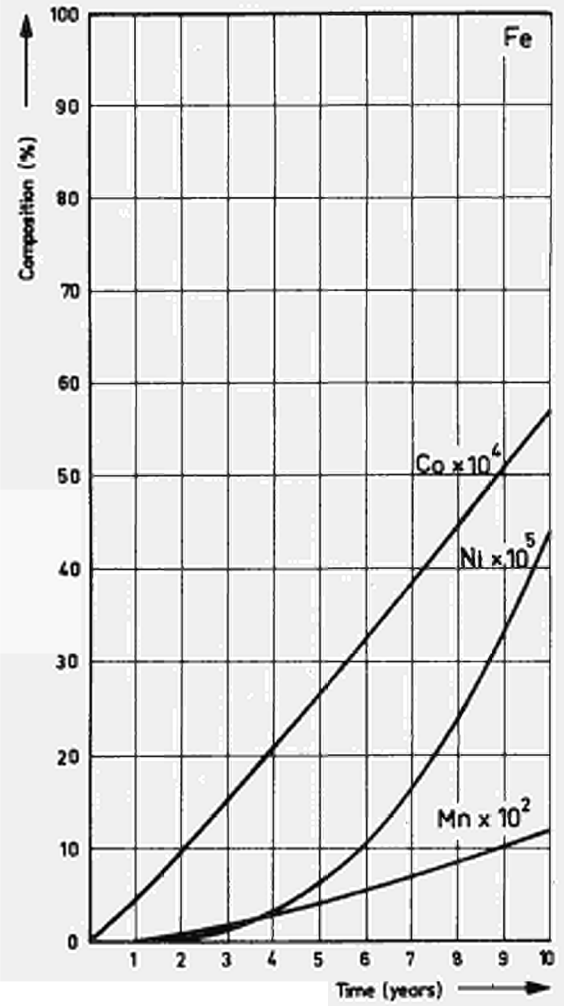


Fig. 6

Iron

Thermal neutron
flux 2×10^{14} n cm^{-2} sec^{-1}

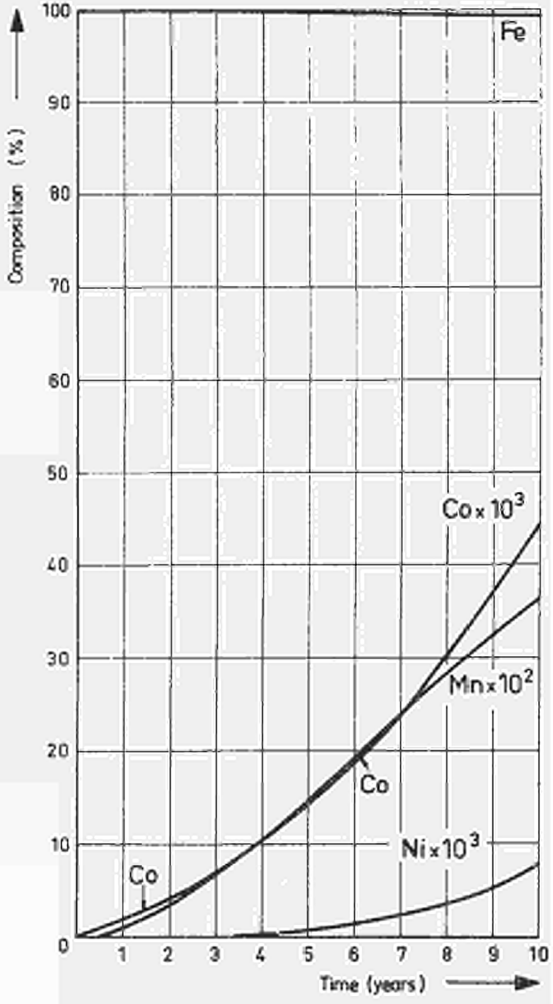


Fig. 7

Iron

Thermal neutron
flux 4.0×10^{14} n cm^{-2} sec^{-1}

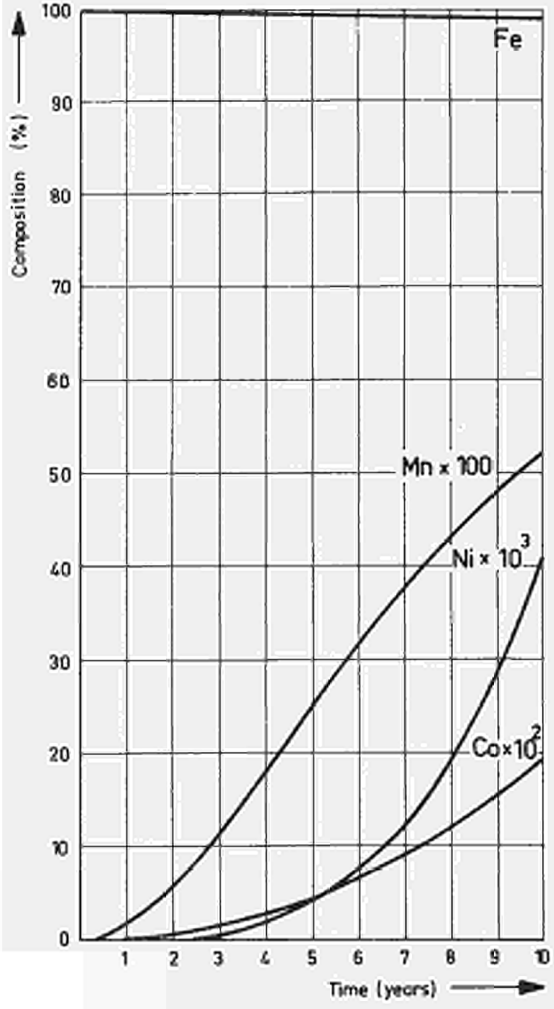


Fig.8

Ru

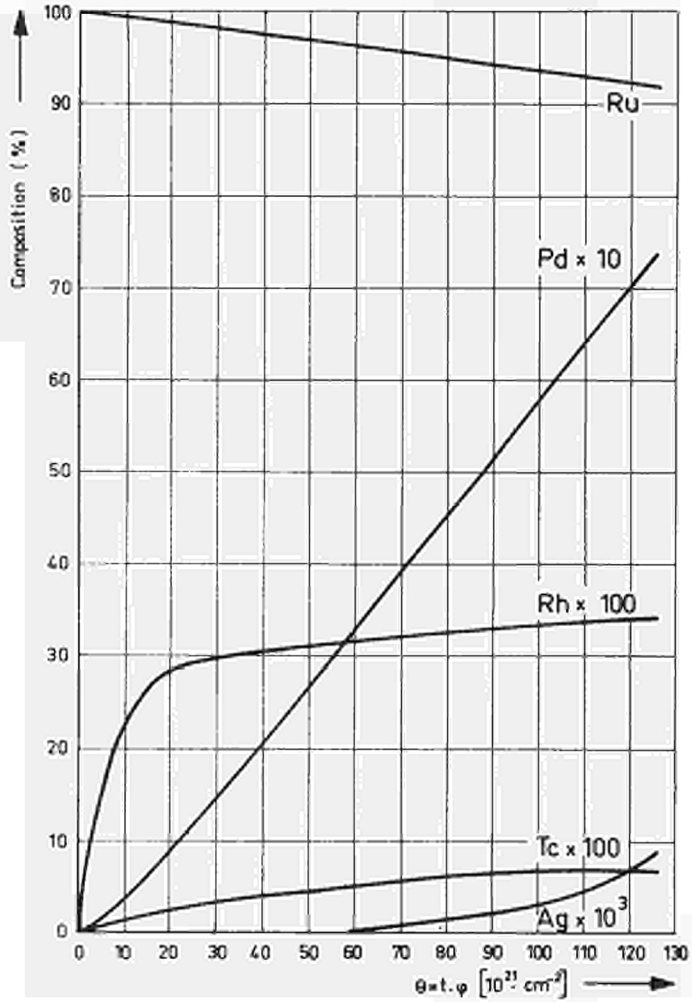


Fig. 9

Pd

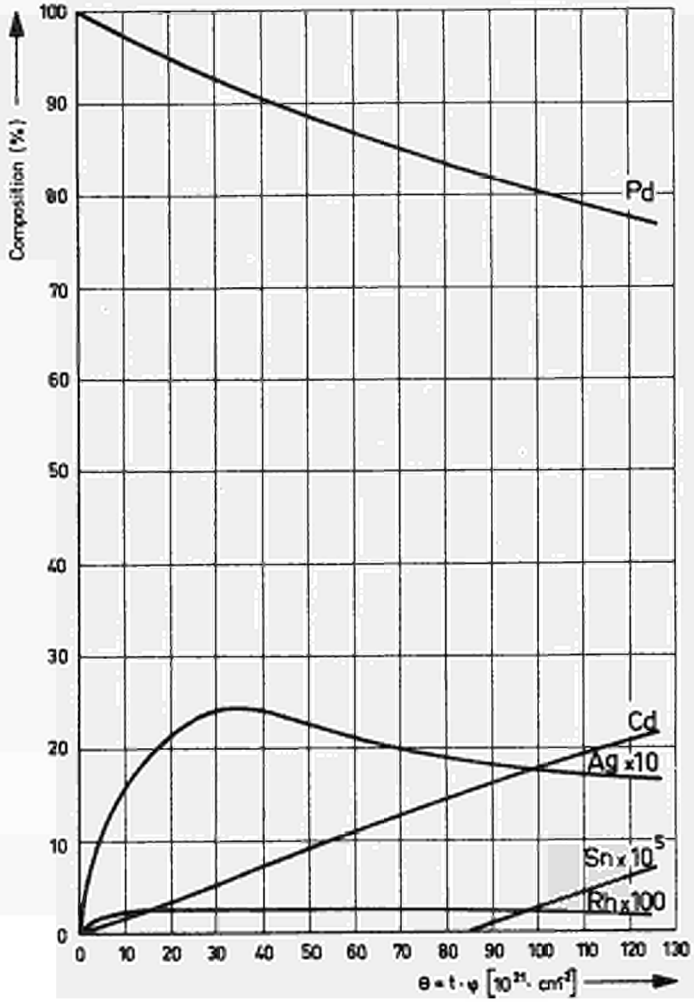


Fig. 10

Nickel

Thermal neutron flux .5 x 10^{14} n cm^{-2} sec^{-1}

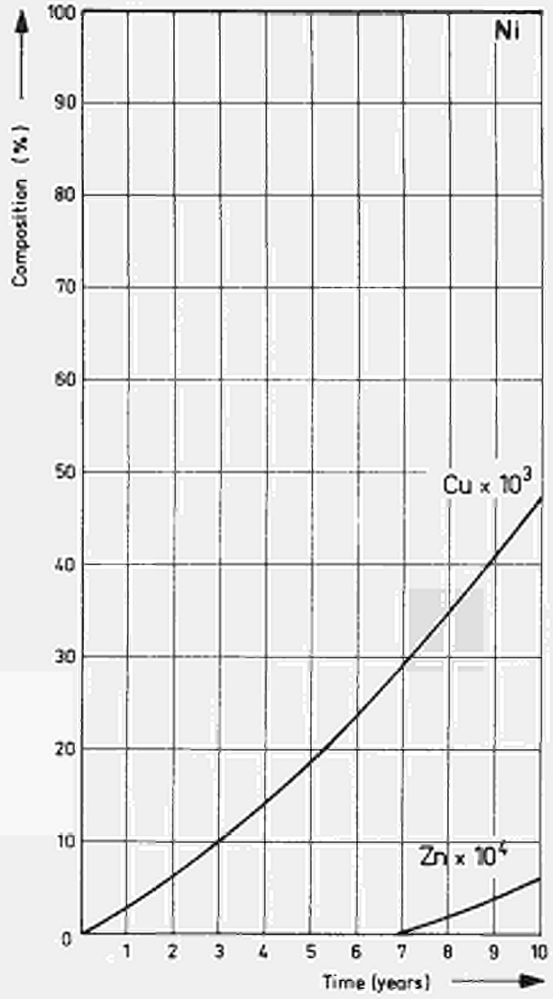


Fig 11

Nickel

Thermal flux 1.0 x10^{14} n cm^{-2} sec^{-1}

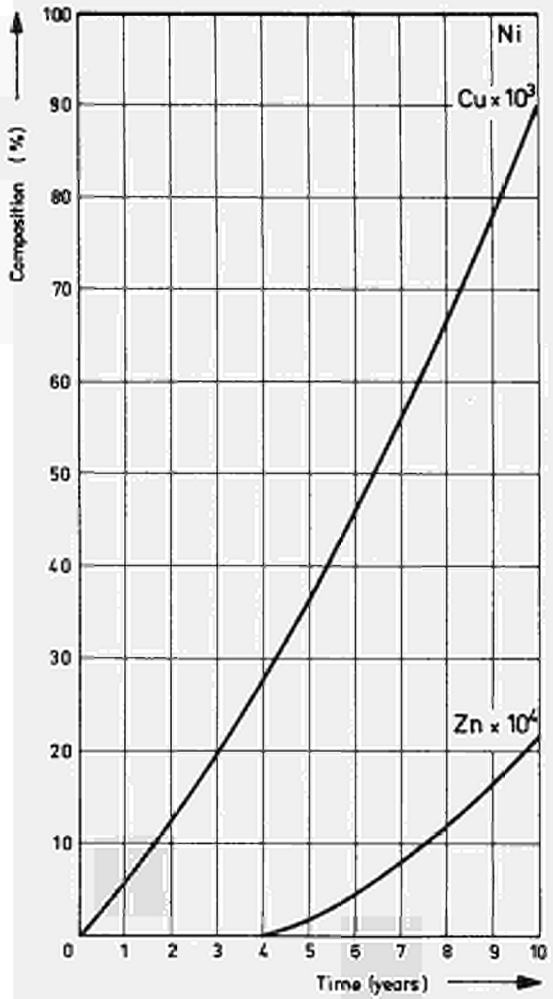


Fig. 12

Nickel

Thermal neutron
flux $2.\times10^{14}$ n cm^{-2} sec^{-1}

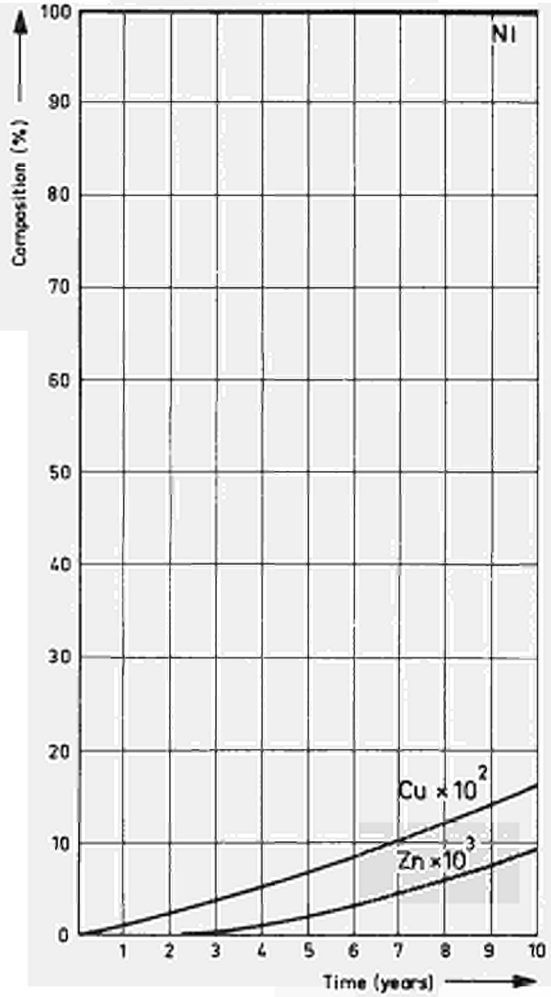


Fig 13

Nickel

Thermal neutron
flux 4×10^{14} n cm^{-2} sec^{-1}

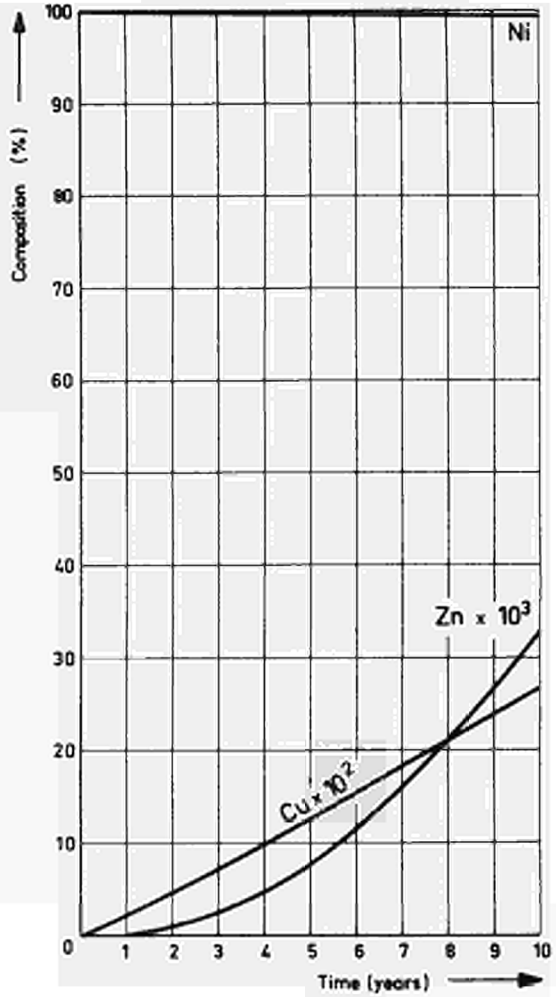


Fig. 14

Pt 90% Rh 10%

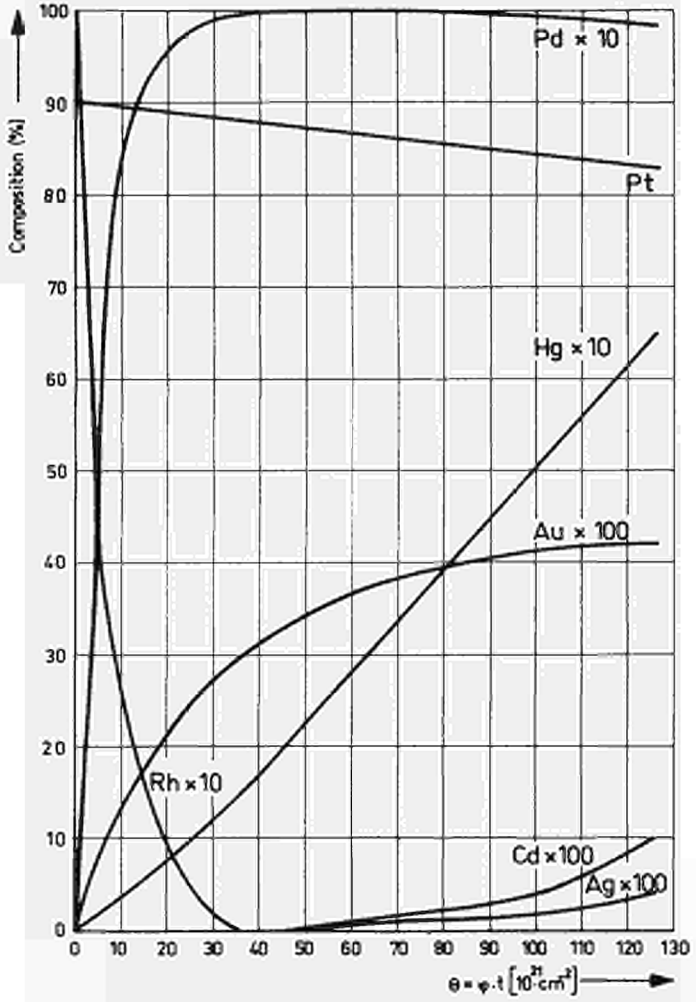


Fig. 15

Pt 99.9% MO 01%

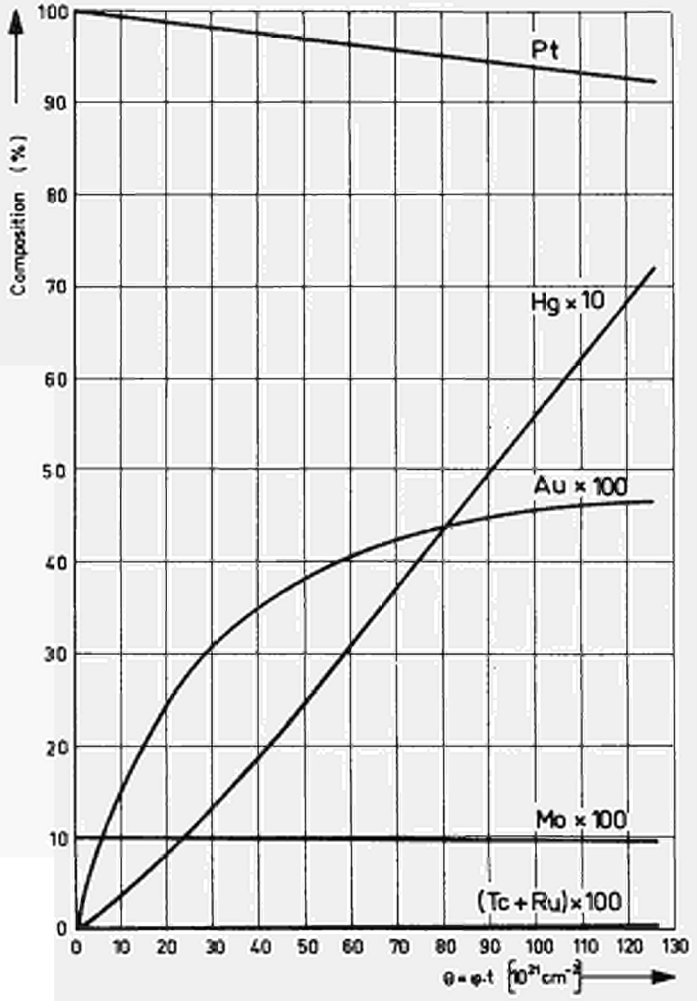


Fig. 17

99.97 Pt + .03 Fe

Thermal neutron flux $.5 \times 10^{14}$ n cm.$^{-2}$ sec.$^{-1}$

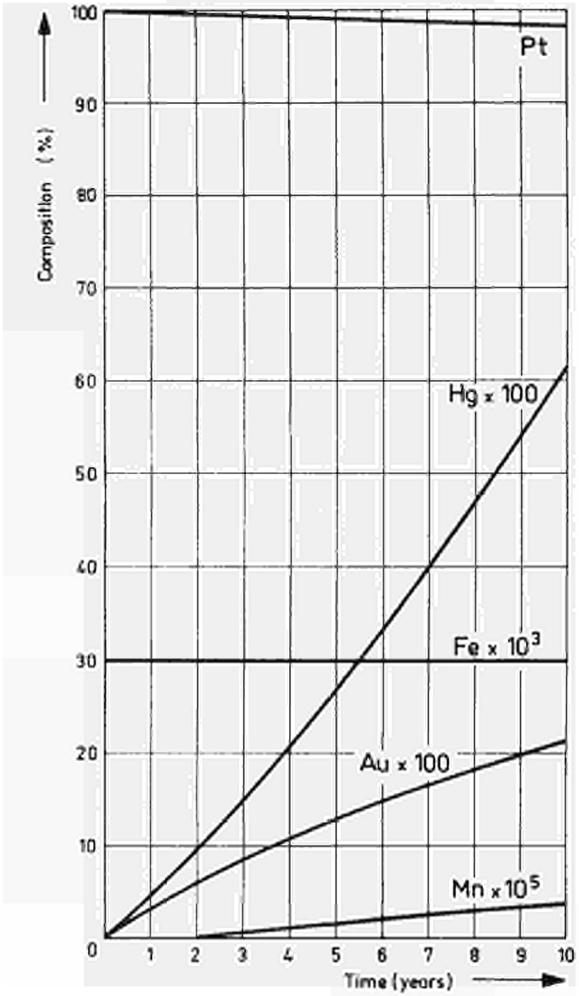


Fig. 16

Pt 95% Mo 5%

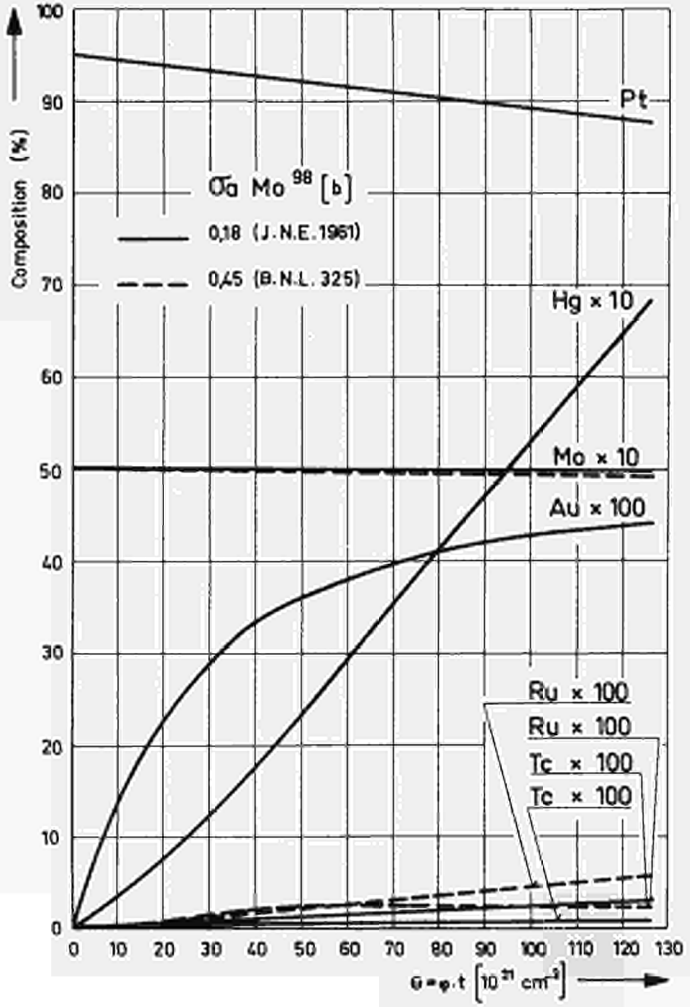


Fig. 18

99.97 Pt + .03 Fe

Thermal neutron flux $1. \times 10^{14}$ n cm^{-2} sec^{-1}

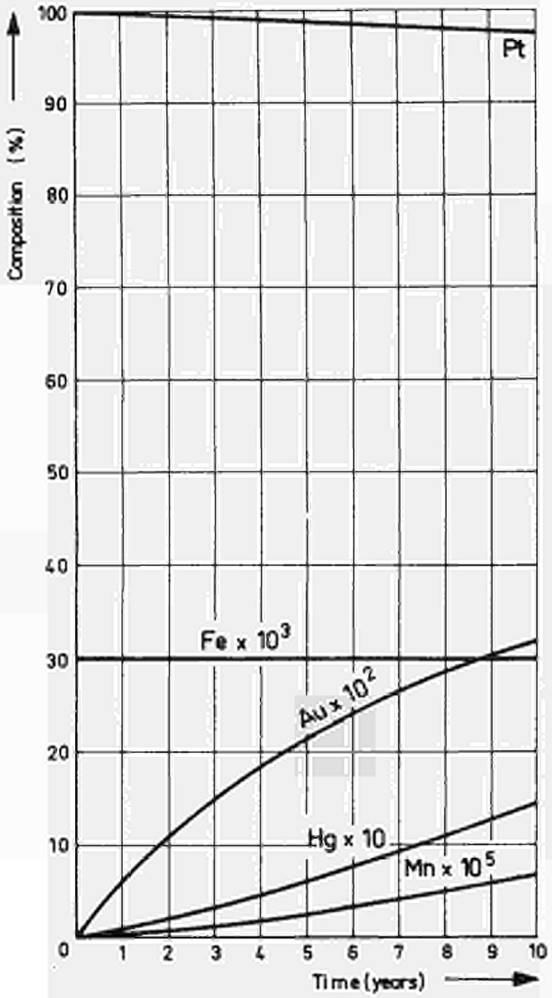


Fig. 19

99.97 Pt + .03 Fe

Thermal neutron flux 2.0×10^{14} n cm^{-2} sec^{-1}

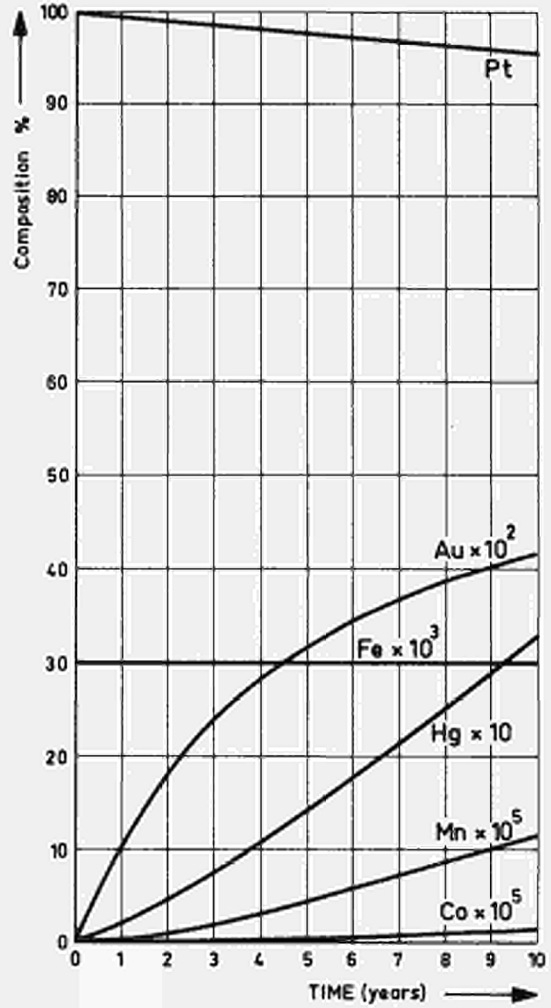


Fig 20

99,97 Pt + .03 Fe

Thermal neutron
flux 4.0×10^{14} n cm^{-2} sec^{-1}

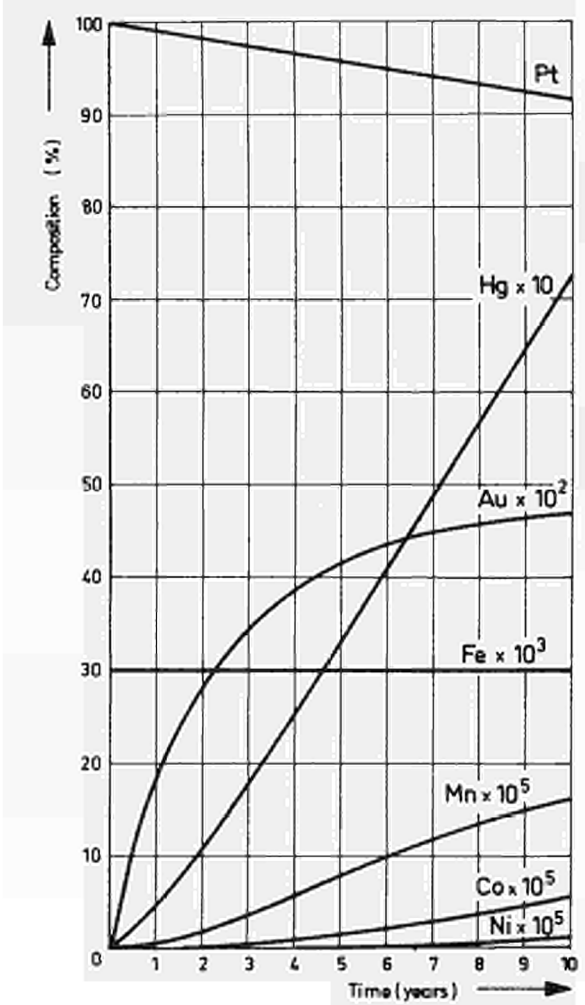


Fig. 21

99.0 Pt + 1.0 Fe

Thermal neutron
flux $.5 \times 10^{14}$ n cm^{-2} sec^{-1}

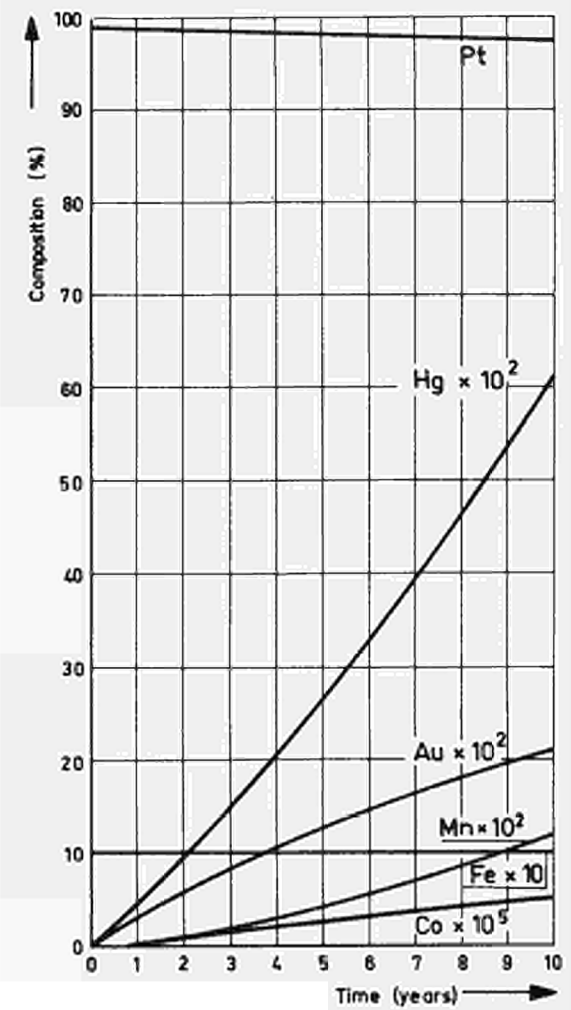


Fig. 22

99.0 Pt + 1.0 Fe

Thermal neutron
flux 1.0×10^{14} n cm^{-2} sec^{-1}

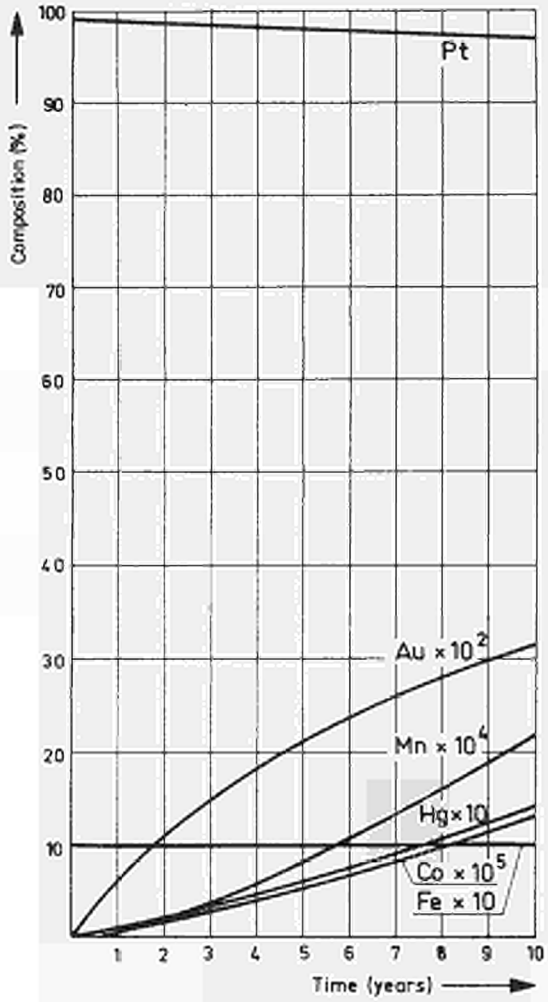


Fig. 23

99.00 Pt + 1.00 Fe

Thermal neutron
flux $2. \times 10^{14}$ n cm^{-2} sec^{-1}

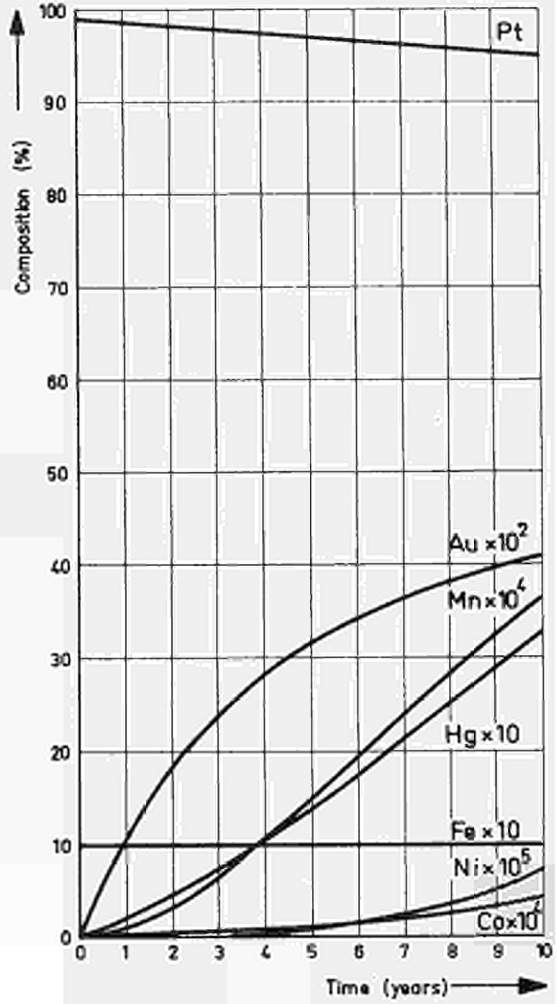


Fig. 24

99.0 Pt + 1.0 Fe

Thermal neutron
flux 4.0×10^{14} n cm^{-2} sec^{-1}

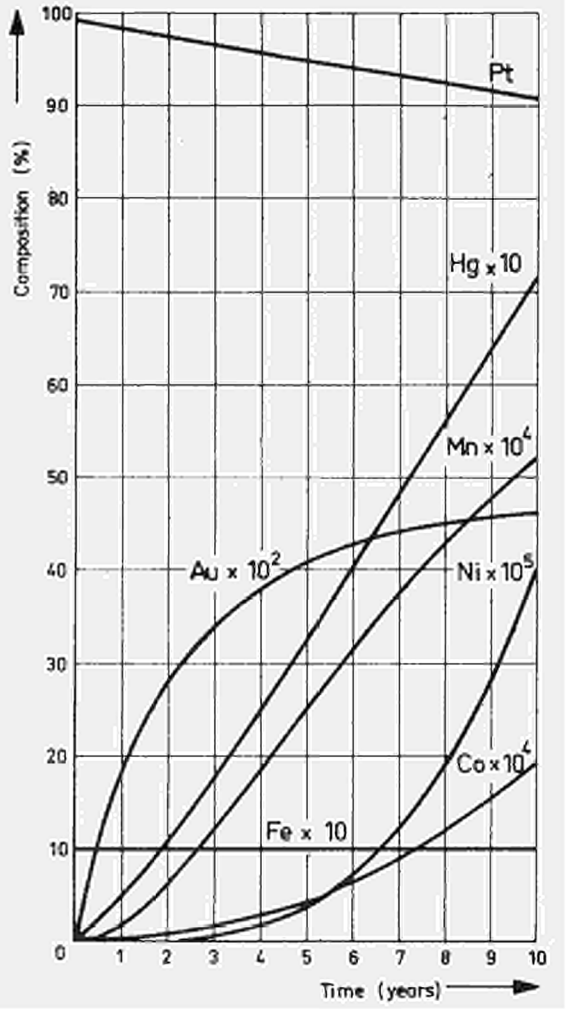


Fig. 25

Pt 99.9 % Ru 01%

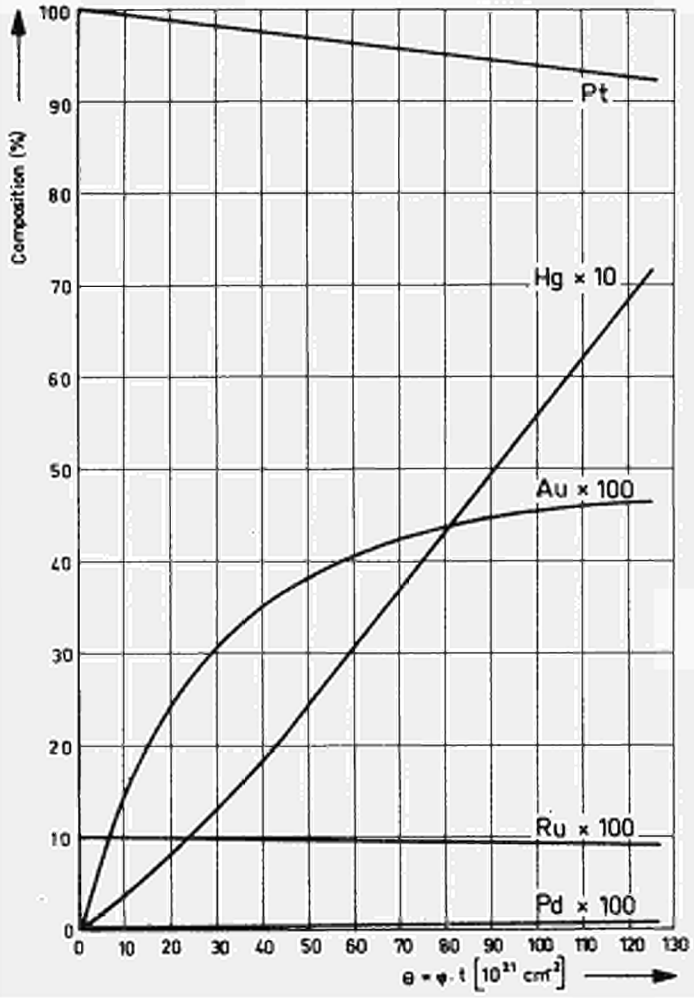


Fig. 26

Pt 96 % Ru 4%

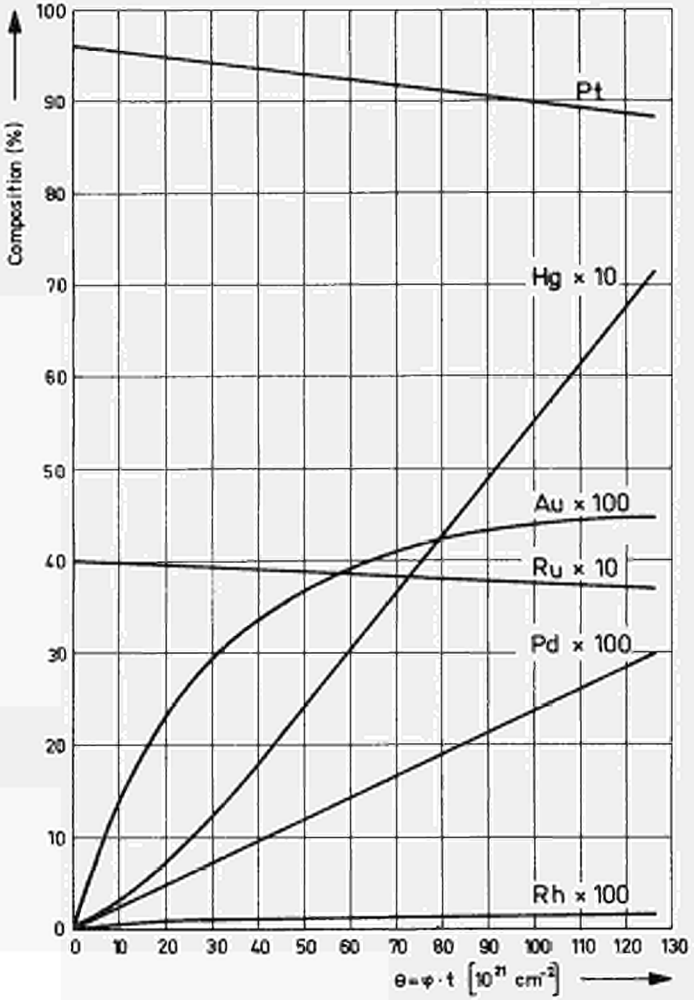


Fig. 27

Pt 99% Pd 1%

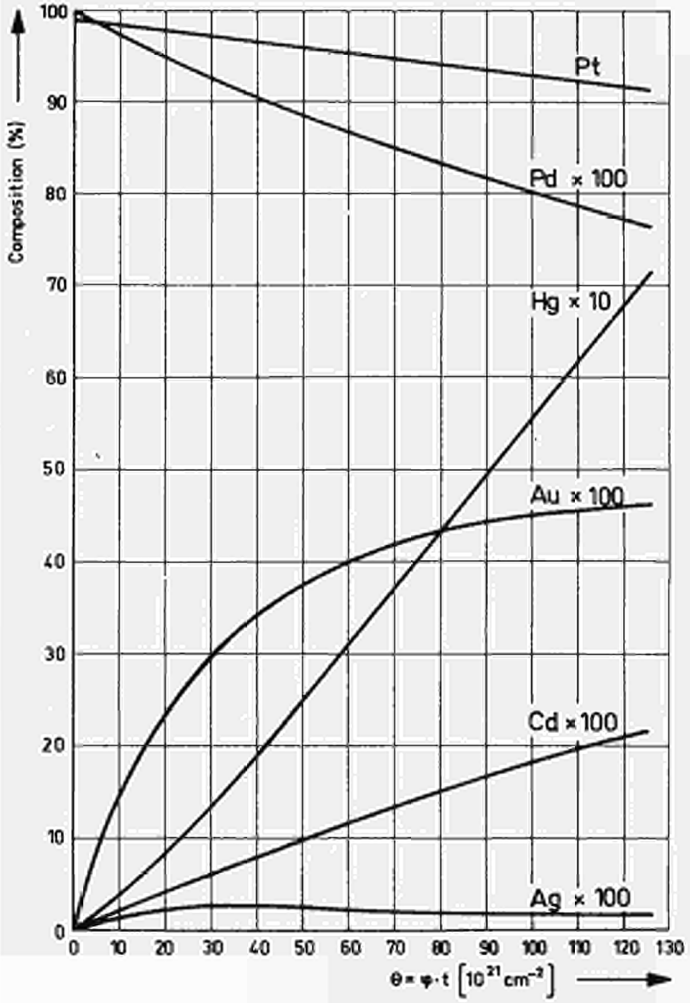


Fig. 28

Pt 80% Pd 20%

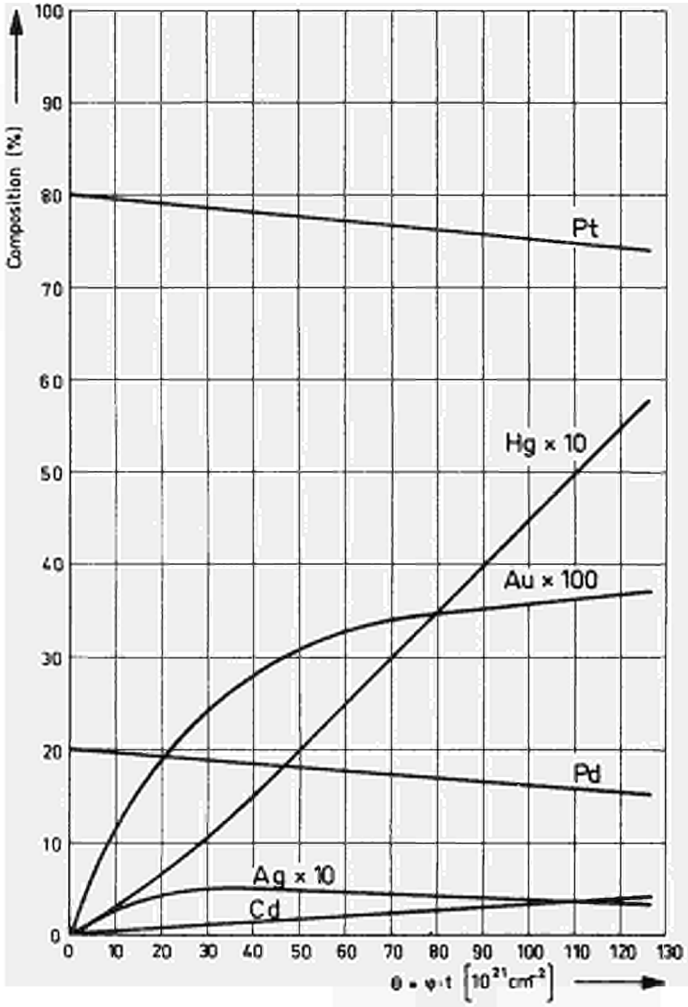


Fig. 29

95,5 Pt+4,5 Ni

Thermal neutron flux 0.5×10^{14} n cm^{-2} sec^{-1}

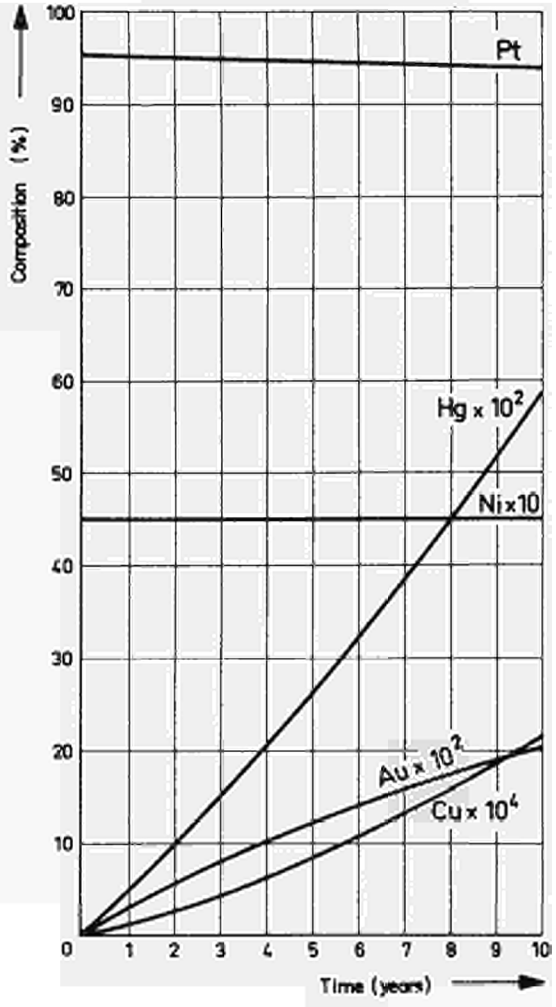


Fig. 30

95.5 Pt + 4.5 Ni

Thermal neutron flux 1.0×10^{14} n cm^{-2} sec^{-1}

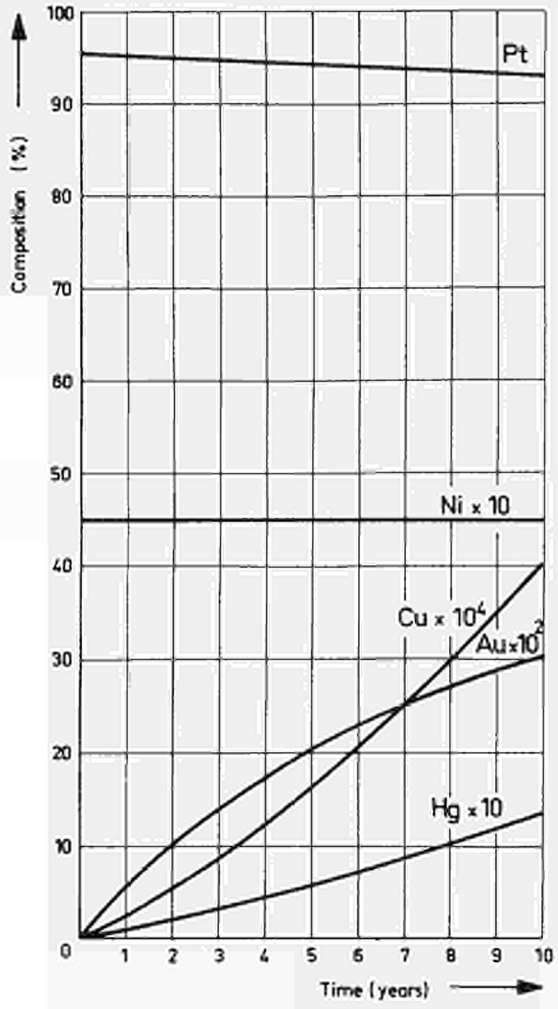


Fig. 31

95.5 Pt + 4.5 Ni

Thermal neutron flux $2. \times 10^{14}$ n cm^{-2} sec^{-1}

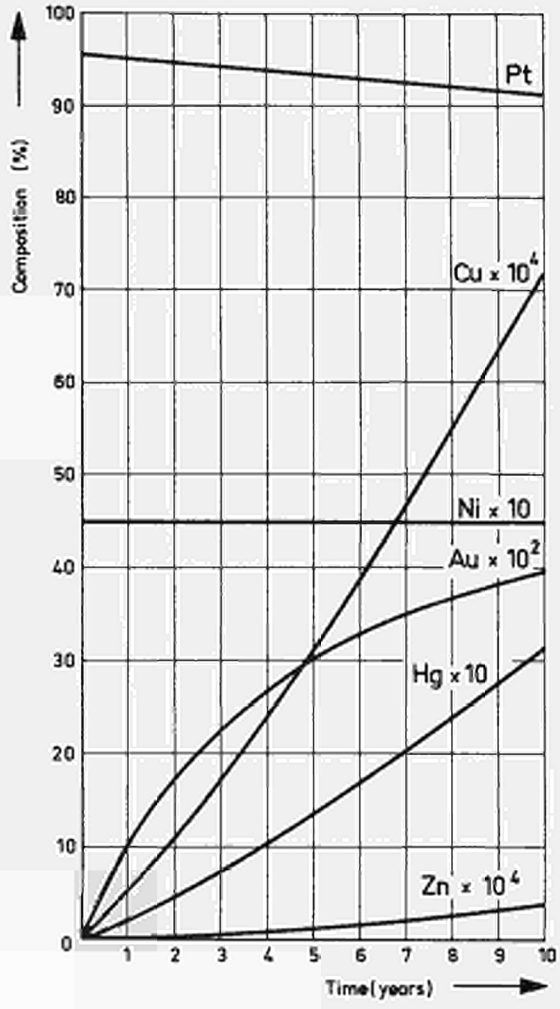


Fig. 32

95.5 Pt + 4.5 Ni

Thermal neutron flux $4.\times 10^{14}$ n cm.$^{-2}$ sec.$^{-1}$

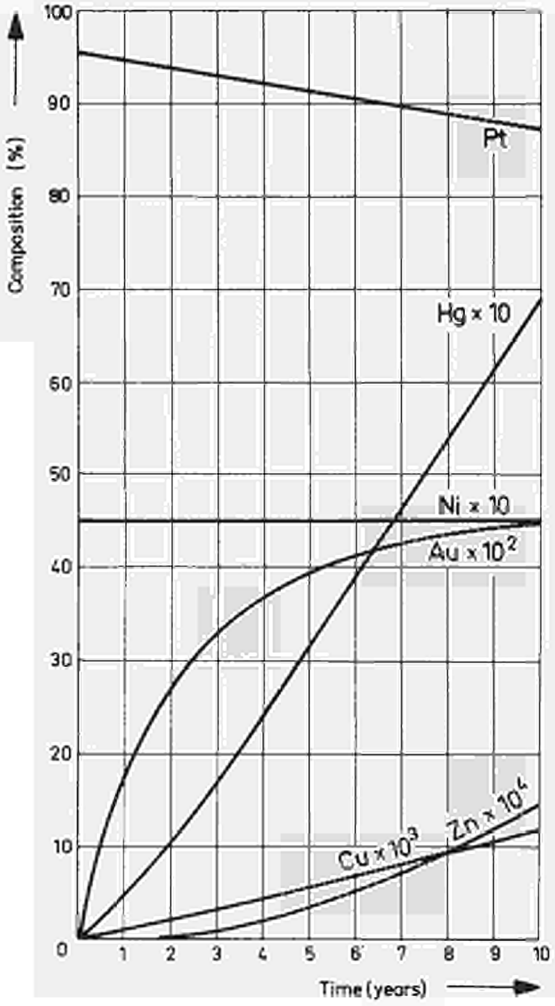


Fig. 33

Pt

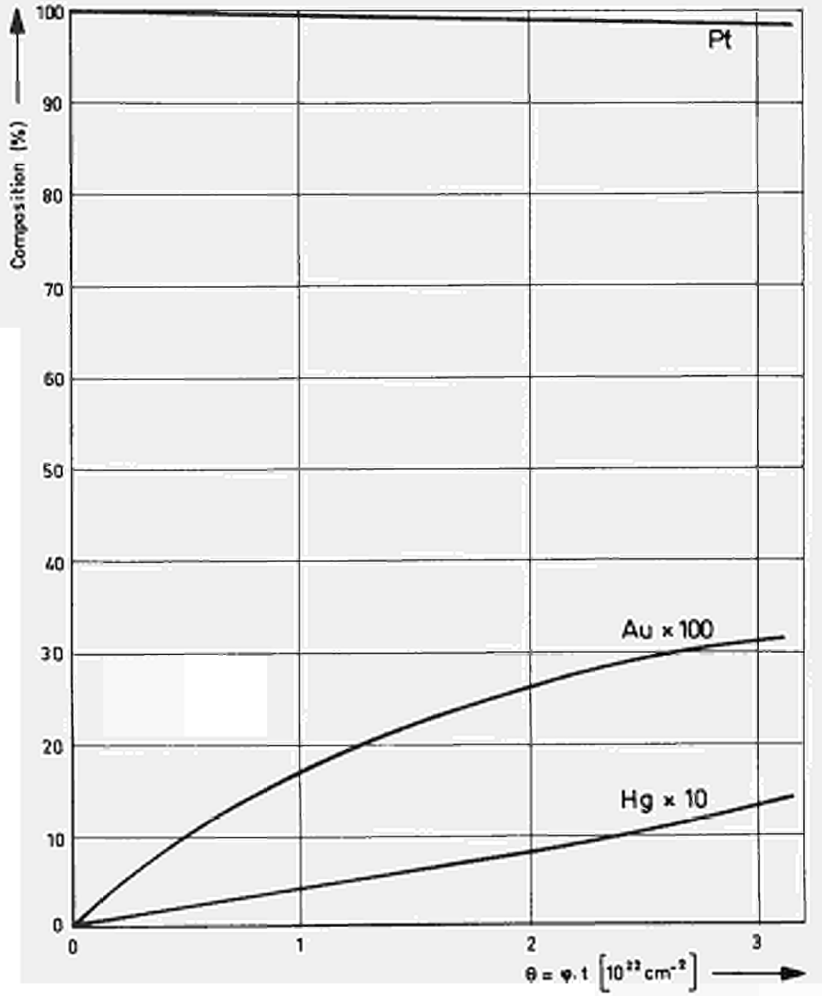


Fig. 34

Pt + 10 % Rh

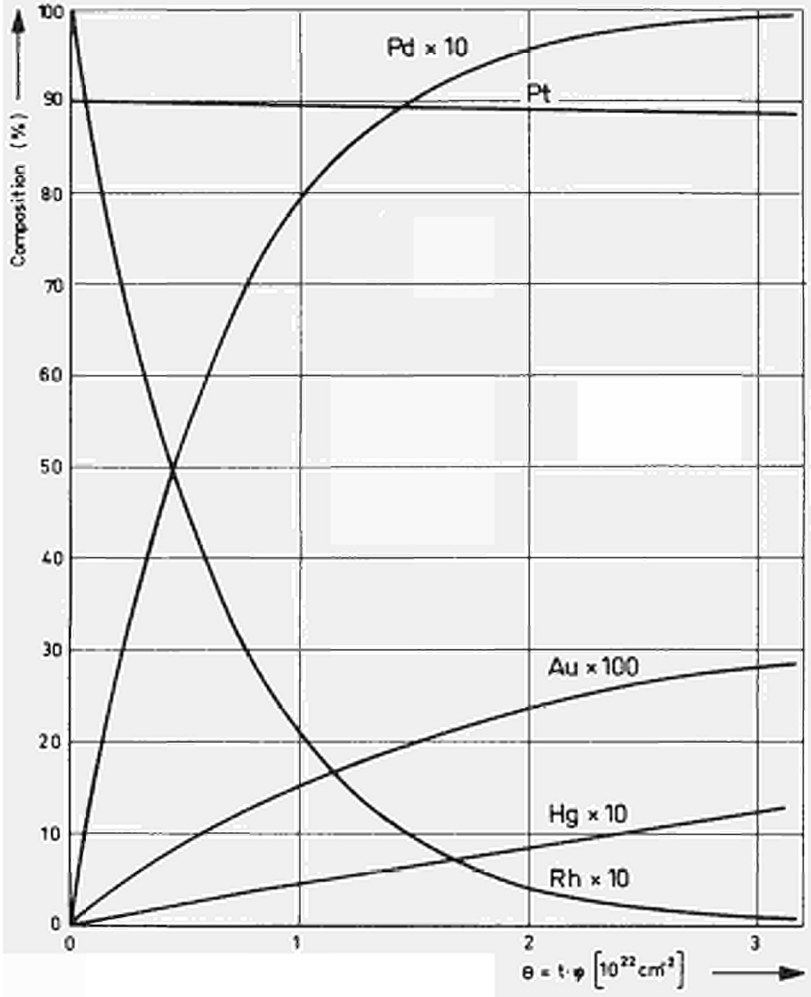


Fig 35

Pt + 0,1% Mo

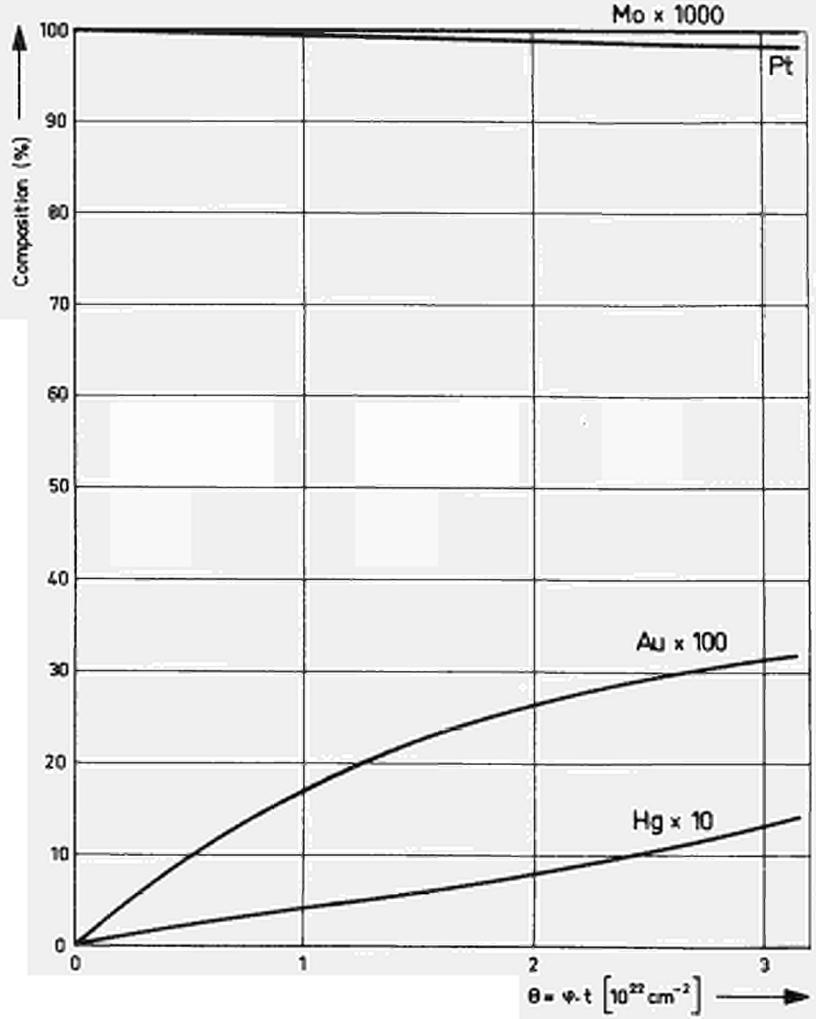


Fig. 36

Pt + 1 % Mo

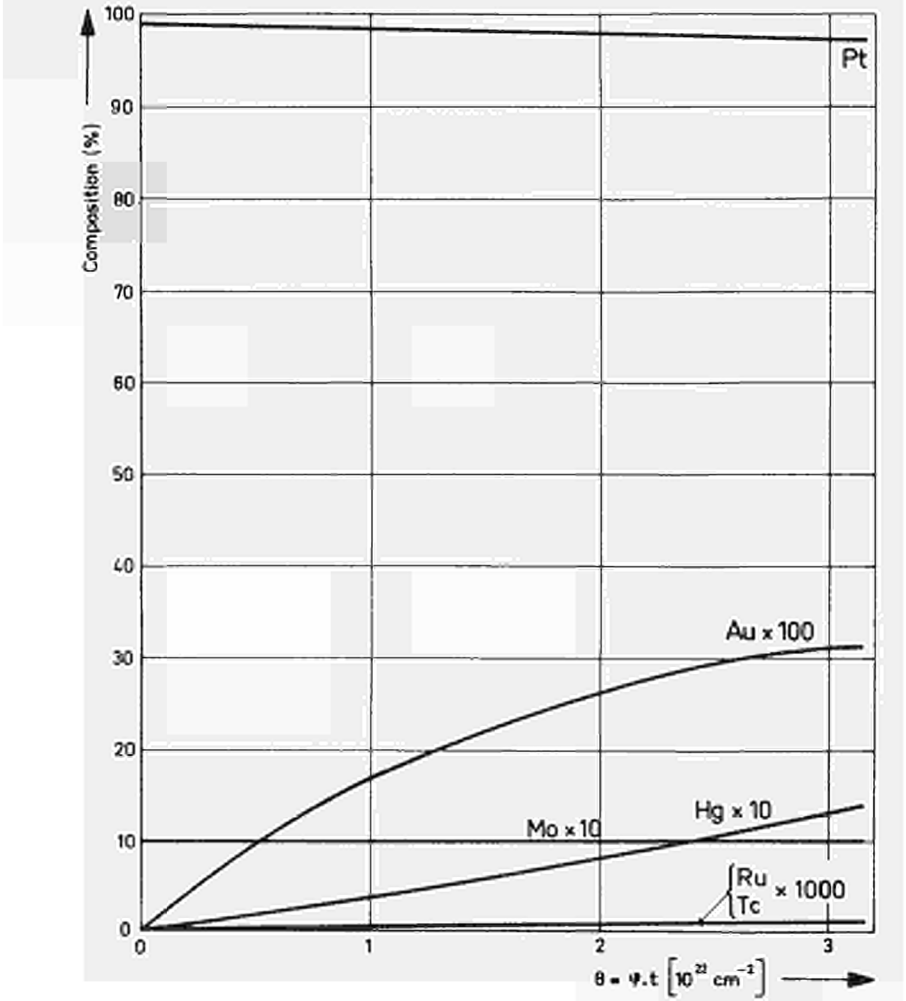


Fig. 37

Pt + 5 % Mo

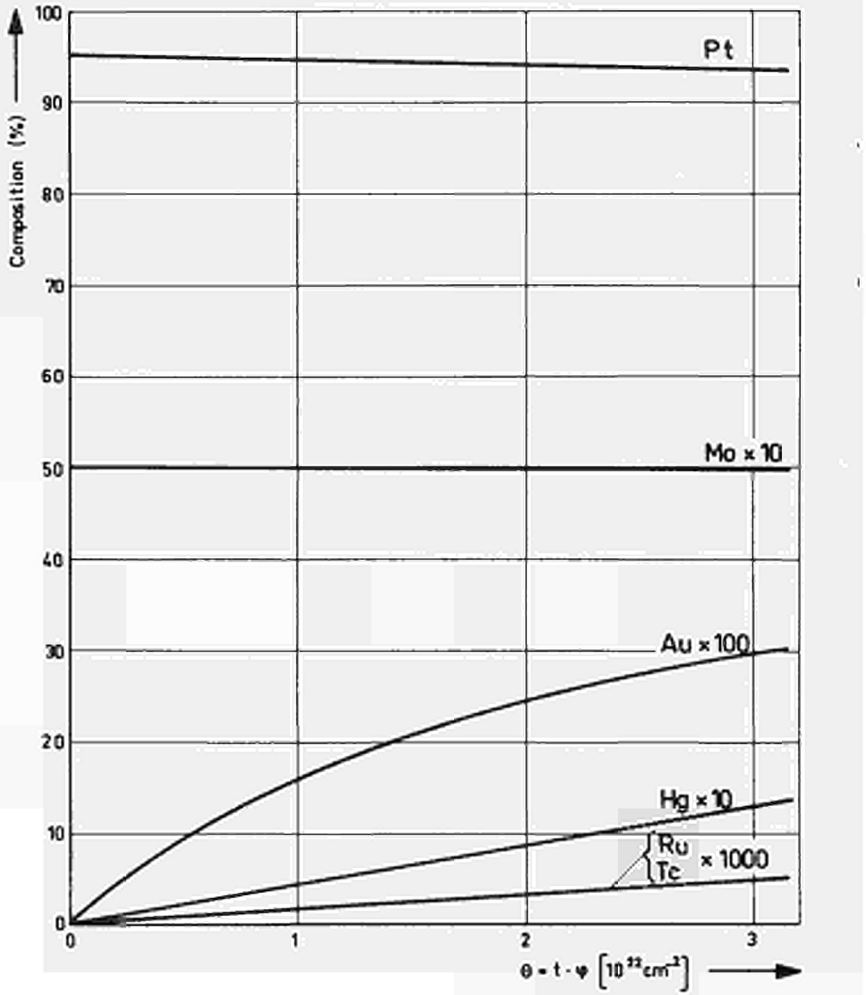


Fig.38

Pt + 1 % Ru

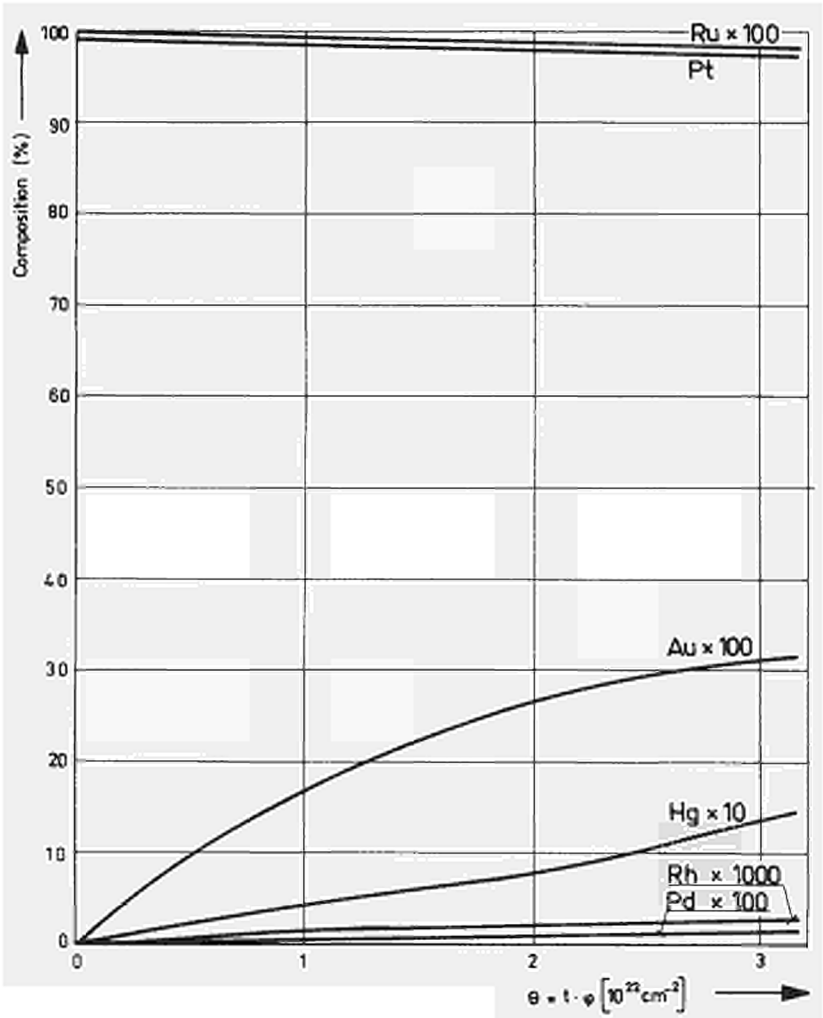


Fig.39

Pt + 2 % Ru

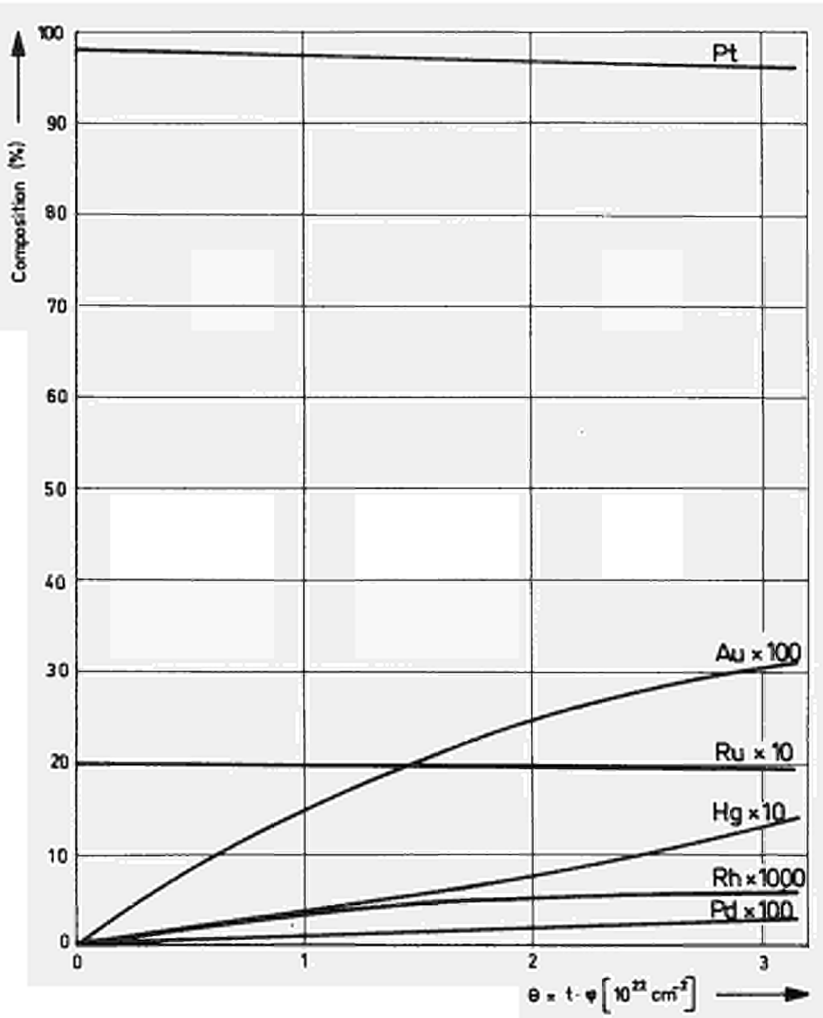


Fig. 40

Pt + 3 % Ru

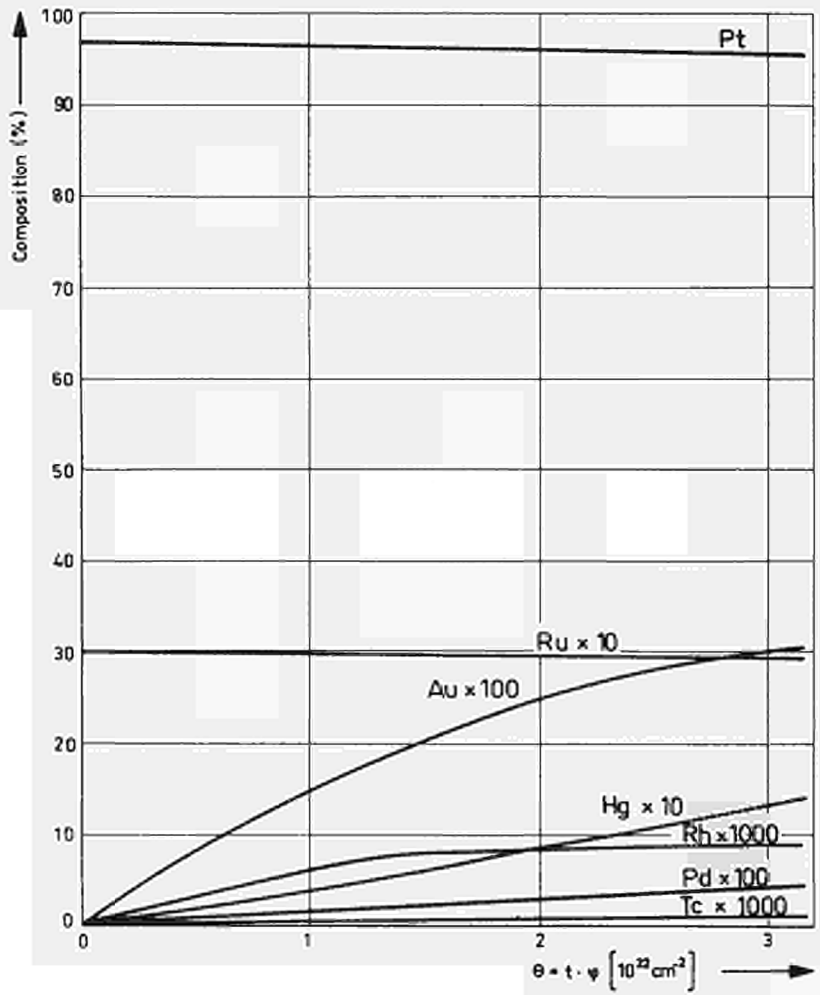
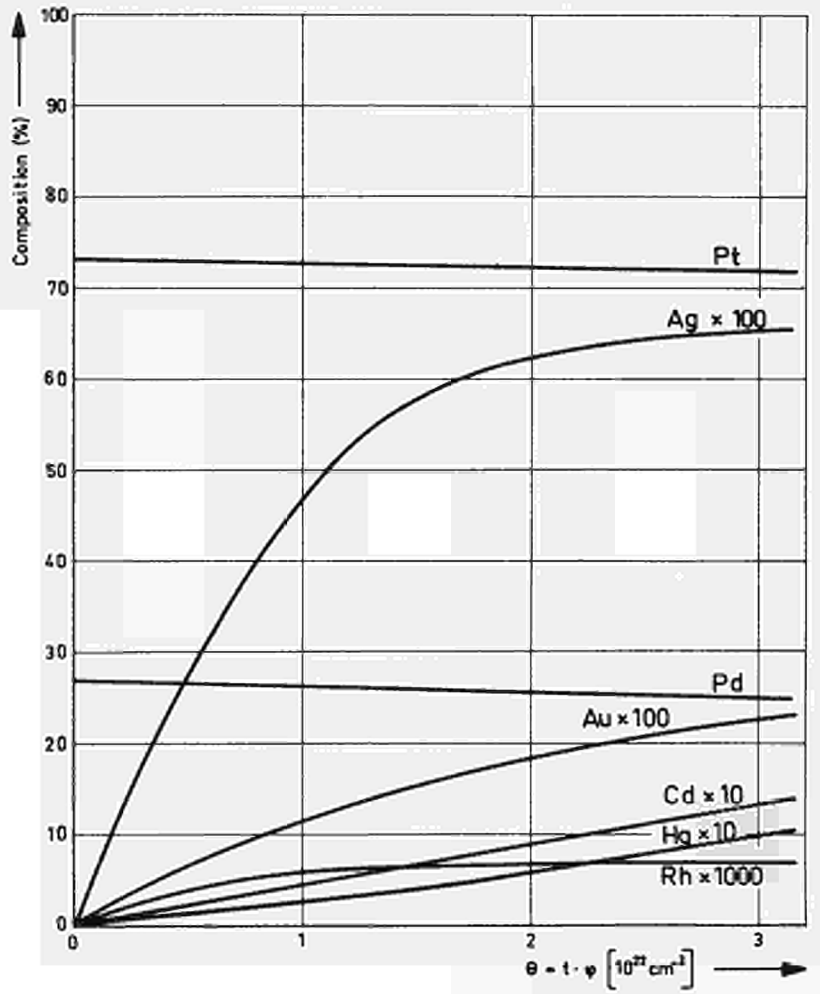


Fig. 41

Pt + 27 % Pd



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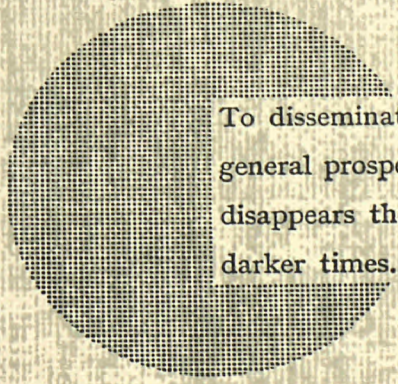

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Alfred Nobel

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