



KERNFORSCHUNGSANLAGE JÜLICH GmbH

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**NEUTRON IRRADIATION OF
NON-METALLIC FIRST WALL
MATERIALS FOR FUSION DEVICES**

by

K. Koizlik

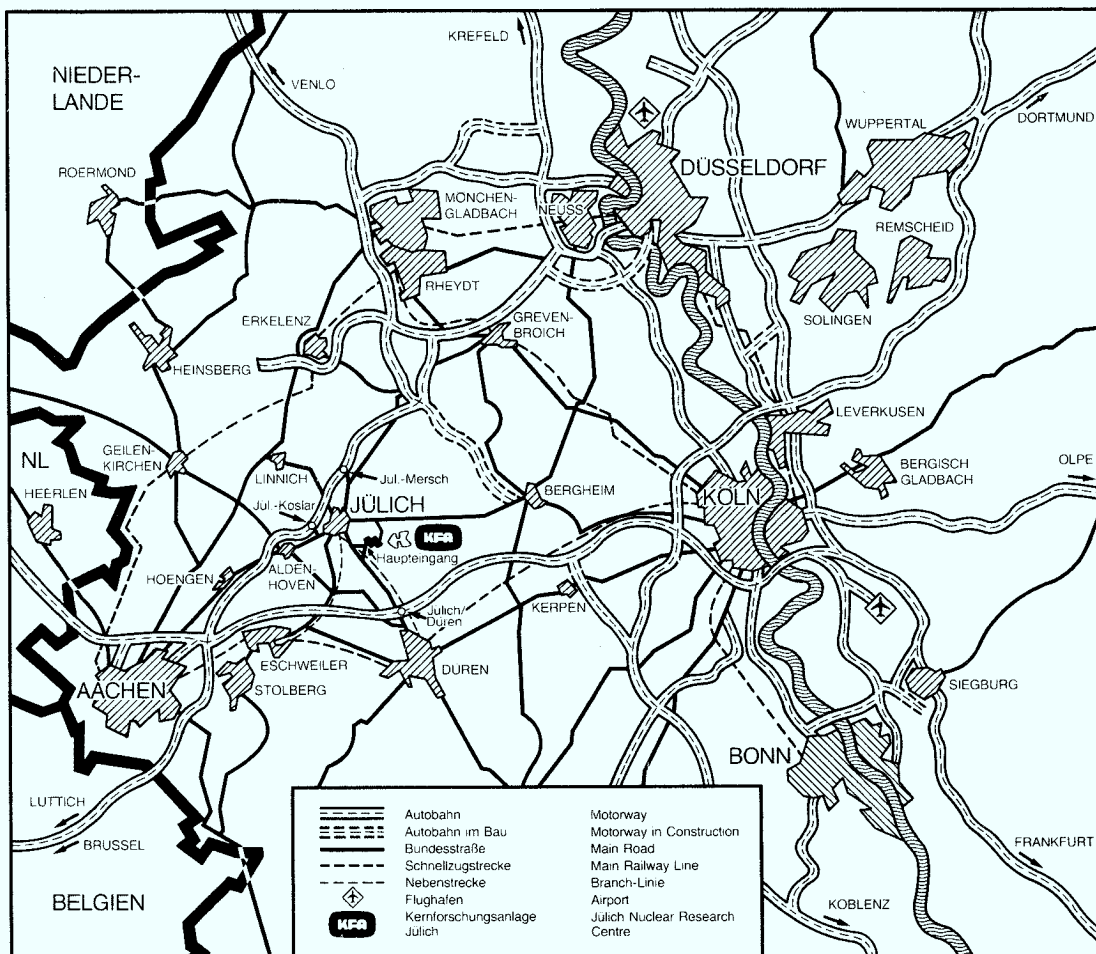
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Jül - 2280
May 1989
ISSN 0366-0885



Als Manuskript gedruckt

Berichte der Kernforschungsanlage Jülich – Nr. 2280

Institut für Reaktorwerkstoffe Jül – 2280

Zu beziehen durch: ZENTRALBIBLIOTHEK der Kernforschungsanlage Jülich GmbH
Postfach 1913 · D-5170 Jülich (Bundesrepublik Deutschland)

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Neutronenbestrahlung Nicht-
Metallischer Erste-Wand
Werkstoffe von Fusionsanlagen

von

K. Koizlik, H. Hoven, J. Linke,
H. Nickel, E. Wallura

Kurzfassung

Im Fusionsreaktor werden etwa 80 % der Fusionsenergie durch die Neutronen der Fusionsreaktion zur ersten Wand transportiert. Aus diesem Grund ist es notwendig, die Neutronen-induzierten Änderungen der Materialeigenschaften und Werkstoffschäden in den Erste-Wand-Materialien zu untersuchen. Da eine Neutronenquelle, deren Energiespektrum und Neutronenfluß den Bedingungen im Fusionsreaktor entsprechen, noch nicht verfügbar ist, hat das IRW ein Bestrahlungsprogramm für nicht-metallische Erste-Wand-Materialien in Spaltungsreaktoren begonnen. Der vorliegende Bericht faßt die fusionsrelevanten Ergebnisse von Bestrahlungsexperimenten zusammen, die während der Entwicklung des gasgekühlten Hochtemperatur-Reaktors durchgeführt wurden, diskutiert die Folgerungen für das neue Bestrahlungsprogramm, dessen Testphilosophie sowie die ersten Bestrahlungstests dieser neuen Versuchsreihe.

Neutron Irradiation of
Non-Metallic First Wall
Materials for Fusion Devices

by

K. Koizlik, H. Hoven, J. Linke,
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Abstract

In ignited fusion plasmas, about 80 % of the fusion energy will be transported to the first wall by an intense flux of energetic neutrons. Therefore the neutron induced changes of material properties and material damages have to be investigated for the first wall materials. Since a realistic fusion neutron source is not available so far IRW has set up a neutron irradiation program for non-metallic first wall materials in nuclear fission reactors. This report summarizes the fusion-relevant irradiation data from irradiation experiments that have been carried out during the development of the High Temperature Gas Cooled Reactor, the consequences for the new irradiation program, the test philosophy derived from these results, and the first irradiation experiments in this new series.

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1. INTRODUCTION

The development of a net-energy producing fusion reactor covers a broad spectrum of basic scientific and technological problems the most important of which will have to be solved before ignition of the fusion process and, moreover, before a demo reactor can be built. One of these central problems is the availability of plasma facing materials, i.e. of materials which form or cover the inner, that means the plasma facing side of the first material wall around the hot plasma.

Parts of this first wall are in direct contact with the hot plasma, namely the high heat flux components limiters and divertors. The larger part of first wall interacts with the plasma normally indirectly by particle and electromagnetic radiation from the plasma. This plasma-wall interaction /1,2/ attacks the first wall materials and is one of the reasons why only few materials may be acceptable for this application. The other reason is the erosion of the materials /3,4/ and, as a consequence, the contamination of the plasma by at least part of the eroded material.

In future plasma devices and, in particular, in a fusion reactor, the neutrons play an important role in the plasma wall interaction. The reason for this fact is that irradiation of crystalline material with energetic neutrons degrades many of the important material parameters and causes dimensional changes of the materials. This is in particular true for carbon based materials which are at present the most promising candidates for the plasma facing side of the first wall. Neutron irradiation tests have thus become an important task for the development and characterization of first wall materials.

2. PLASMA WALL INTERACTION

The hot fusion (-near) plasma interacts with the first materials wall thus giving rise to a mutual affection of the material and

the plasma. From the materials point of view, plasma wall interaction is a term summarizing a group of individual processes /5,6/ that affect primarily the surface or surface-near parts of the plasma facing material.

The surface-related plasma-wall interaction occurs with different intensity depending on the operational state of the plasma. During "normal" plasma operation, the plasma facing materials are affected in a way that may be tolerable at least for advanced materials. Transient or instable plasma states, on the other hand, can cause, in the worst case, the destruction of the materials and thus the failure of the respective component.

Moreover, the surface-related processes are necessarily coupled to the technical concepts of particle exhaust - primarily He, the "ash" of the fusion process, and impurities -, namely pumped limiters /7/ or divertors /8/. They thus occur with the highest intensity on the surface of these so-called high-heat-flux components. Additionally, they are necessary to extract energy from the plasma, at least as long as the plasma does not reach ignition, and therefore the flux of energetic fusion neutron is not worth mentioning.

But already in plasma devices of the next generation like NET /9/ (Next European Torus) or ITER /10/ (International Thermo-nuclear Experimental Reactor) fusion neutrons and their effects on the first wall will no longer be negligible. This type of plasma-wall interaction has to be seen under two aspects: In an ignited fusion device, about 80 % of the fusion energy is transported by the energetic neutrons and transferred to the first wall and finally to the coolant. This process is no longer related to the surface but extends far into the bulk of the material. On the other hand, the energetic fusion neutrons cause material damage and changes in the materials properties which may reduce directly the lifetime of a first-wall component and may additionally intensify other processes of the plasma-wall interaction.

This is why the interaction of first wall materials and fusion neutrons has to be investigated.

3. EFFECTS OF ENERGETIC NEUTRONS IN NON-METALLIC MATERIALS

In the bulk of a crystalline body, a neutron collides at the end of its mean free path with a lattice atom. In the case of non-fissionable material - and only this is relevant for the plasma-facing side of the first wall - two basic processes can occur, namely the displacement of the lattice atom and the generation of transmutation products.

In non-metallic material, such as carbon based materials or heat-conducting ceramics, Helium and Hydrogen are important transmutation products. They are produced by (n, α) - or (n, p) -reactions and can cause embrittlement of the materials. In particular, for some low-Z elements like carbon the transmutation reactions occur above an energy threshold in the MeV-range, and then their cross sections may show a pronounced energy dependence. This can be seen from fig. 1 /11/ for the elements Be, Al and Si for the (n, α) - and for the (n, p) - and (n, d) -reaction, resp. At energies above about 6 MeV, also C shows a (n, α) -reaction, fig. 2 /11/. The (n, p) -reaction for this element seems to be negligible. The total cross section for the C (n, α) Be - together with the C (n, n') 3α -reaction cross section - is relatively large and reaches about 900 mb /12/ at 14 MeV.

But as far as it can be assumed today, the effects of the gaseous transmutation products should not be as strong as the effects of lattice atom displacements, at least in the case of carbon based materials like graphites and carbon fiber reinforced carbons (CC). This may be true particularly because of the density of these materials which reaches only about 80 % of the theoretical value, the open porosity and, at least for most of the considered first wall components, the elevated temperature of the materials. Nevertheless, this assumption has to be

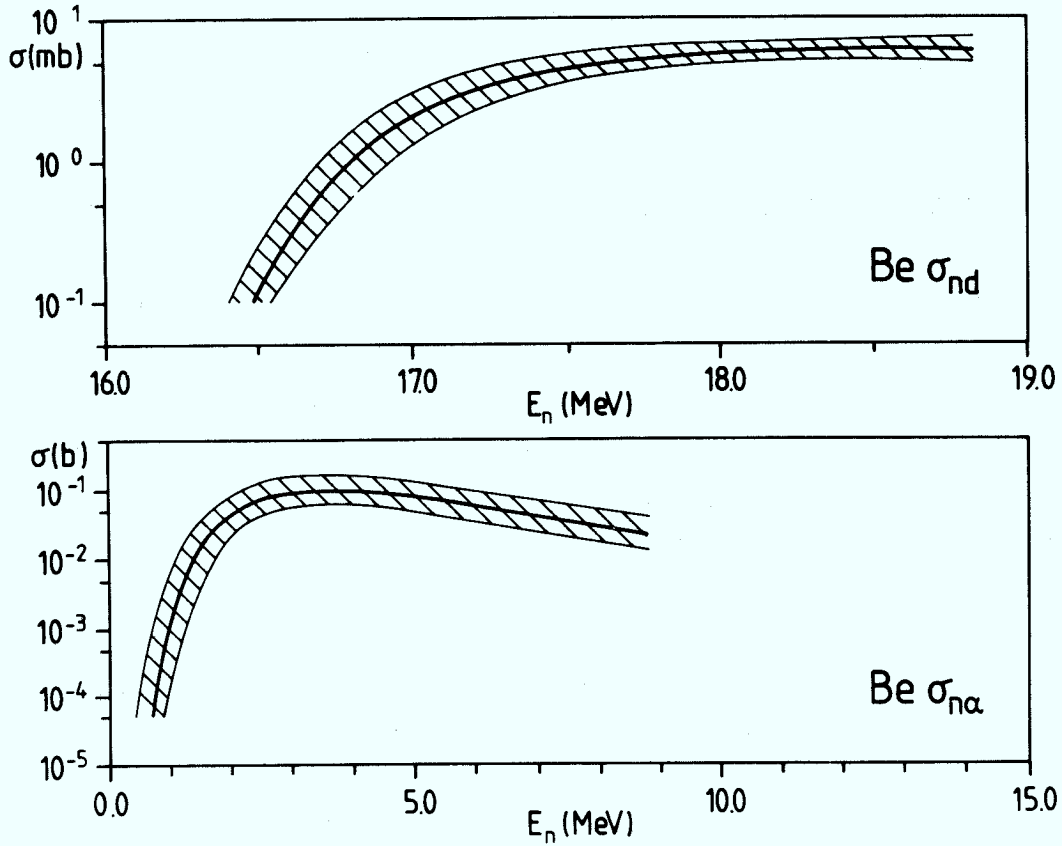


Fig. 1: Energy dependence of transmutation reactions: $\text{Be}/\sigma_{n,d}$ and $/\sigma_{n,\alpha}$ (from Ref. 11); Fig. 1 cont.

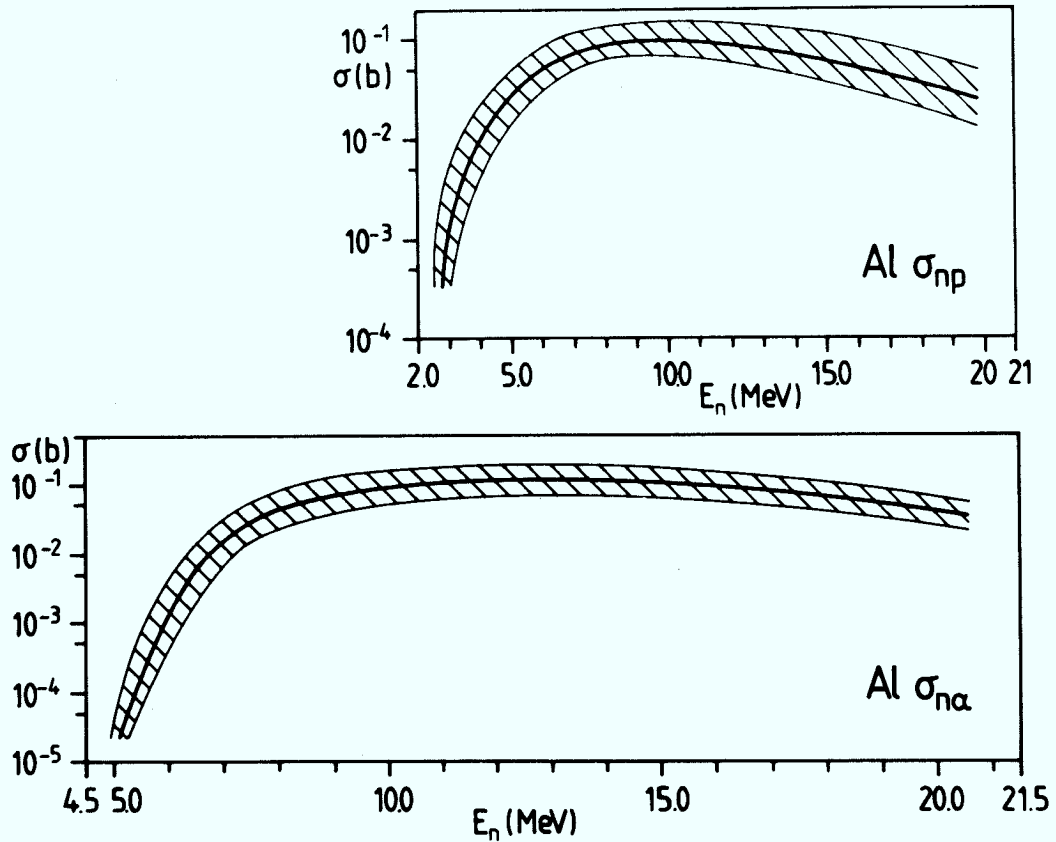


Fig. 1: continued: $\text{Al}/\sigma_{n,p}$ and $/\sigma_{n,\alpha}$; Fig. 1 cont.

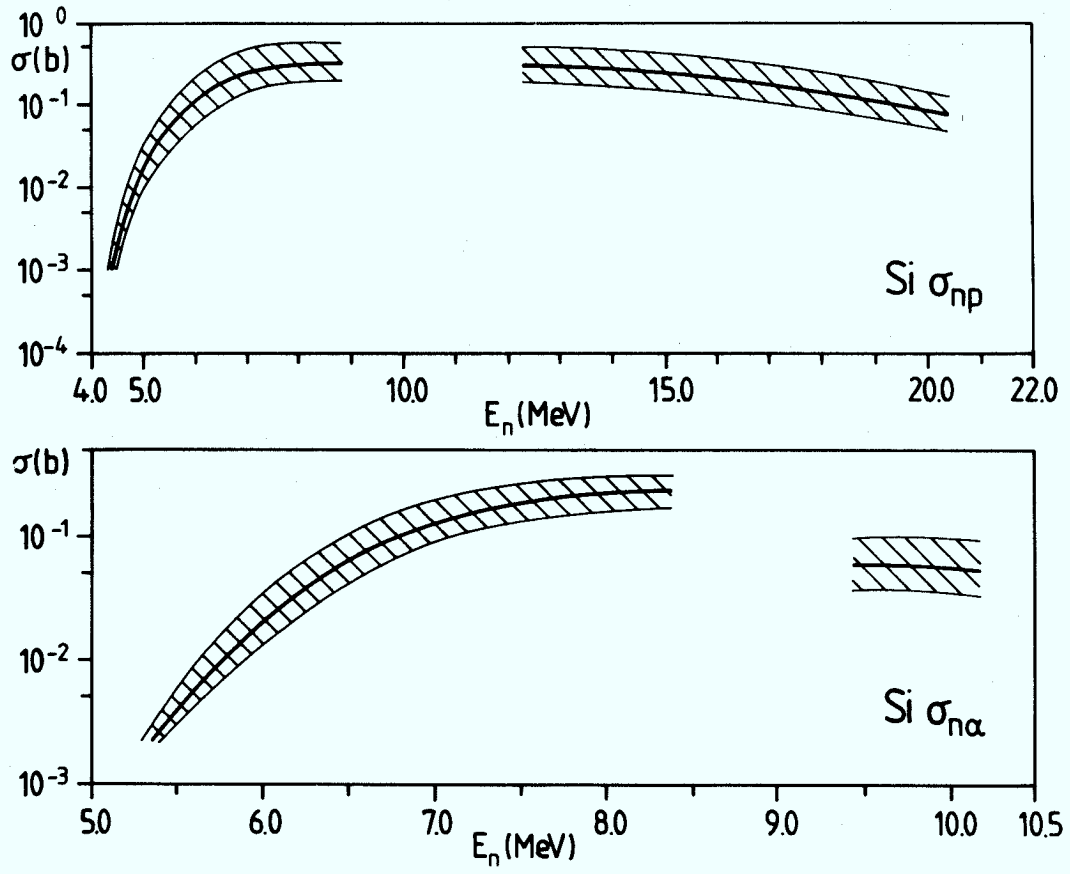


Fig. 1: continued: Si/ $\sigma_{n,p}$ and / $\sigma_{n,\alpha}$

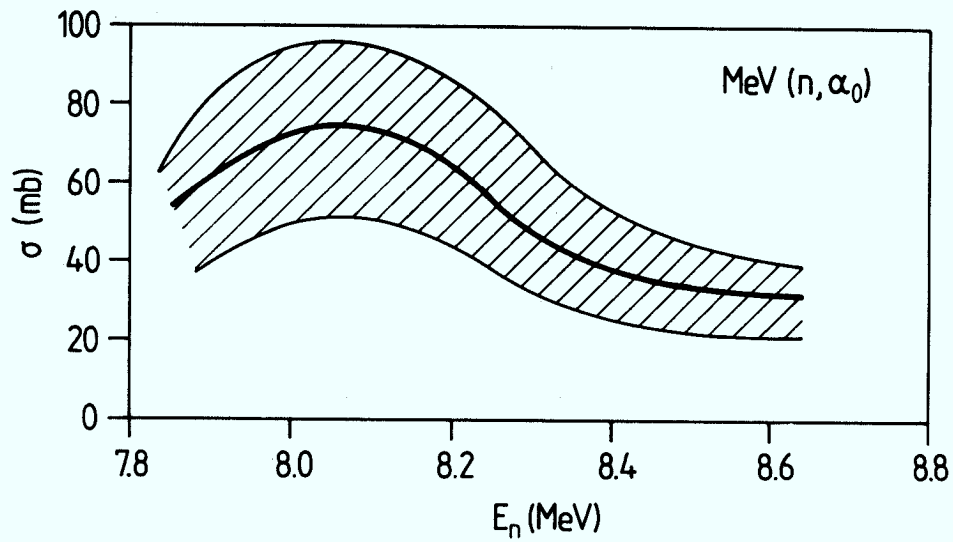


Fig. 2: Energy dependence of the (n, α) -cross section for Carbon 12 (from Ref. 11)

examined in the next future with respect to the relevance of fusion oriented neutron irradiation experiments in fission reactors.

3.1 Irradiation Effects in Graphites

The elastic and inelastic neutron scatter in a graphitic material which causes lattice atom displacements can lead to a strong degradation of material properties, depending on the irradiation temperature. The influence of neutron irradiation on the values of

- thermal conductivity,
- thermal expansion,
- modulus of elasticity,
- strength,

is of particular interest, together with the neutron induced

- dimensional changes, and
- irradiation creep.

These effects of energetic neutrons in graphitic materials have been widely studied during the development of High Temperature Gas-Cooled Reactors (HTGR) for the application of graphites in this type of fission reactor. Thus, a broad data base exists /13,14,15/ for a huge variety of nuclear grade graphites and graphitic matrix materials and their behaviour under neutron impact, but only for a fission neutron spectrum.

Some fundamental informations shall be summarized on the neutronic behaviour of graphitic materials which in principle should be also valid in a fusion neutron spectrum, i.e. in the case of D-T-fusion reactions in a spectrum with neutron energies up to 14 MeV, at least "semi-quantitatively".

3.1.1 Thermal Conductivity

A highly important parameter for the applicability of nuclear graphites in fusion devices is the thermal conductivity. The

value of the thermal conductivity is on the one hand a function of the temperature; it decreases with increasing temperature /13,16/, fig. 3.

The values of thermal conductivity typically lie between 50 and well above $150 \text{ Wm}^{-1} \text{ K}^{-1}$ at room temperature and reach about $50 \text{ Wm}^{-1} \text{ K}^{-1}$ at 1000°C and about $40 \text{ Wm}^{-1} \text{ K}^{-1}$ at 2000°C . For comparison: Cu has a thermal conductivity of $360 \text{ Wm}^{-1} \text{ K}^{-1}$, Mo $140 \text{ Wm}^{-1} \text{ K}^{-1}$, and Ni- and Fe-base alloys of about $30 \text{ Wm}^{-1} \text{ K}^{-1}$ at room temperature.

Irradiation of graphitic materials with energetic neutrons, on the other hand, causes a reduction of the value of thermal conductivity already at constant temperature. This reduction is the smaller, the higher the irradiation temperature, and increases with neutron dose, fig. 4.

The decrease of thermal conductivity is very rapid at the beginning of an irradiation, already at neutron doses below 10^{24} m^{-2} , particularly at low irradiation temperatures, and reaches a factor of about 0,25 at temperatures about or lower than 600°C , but only a factor of 0,5 or even higher about or above 1400°C . The absolute values depend, of course, on the type of material, but the statement is in principal valid for all graphite qualities investigated so far.

An annealing of the irradiated material at temperatures in the range of or higher than the irradiation temperature for several hours can cause, at least partially a healing of the irradiation induced lattice defects and thus a re-increase of the thermal conductivity, although the original values could not be reached in the resp. experiments /13/.

3.1.2 Thermal Expansion

The second important parameter of the graphitic materials for plasma facing components is the coefficient of thermal expansion and its variation by neutron irradiation.

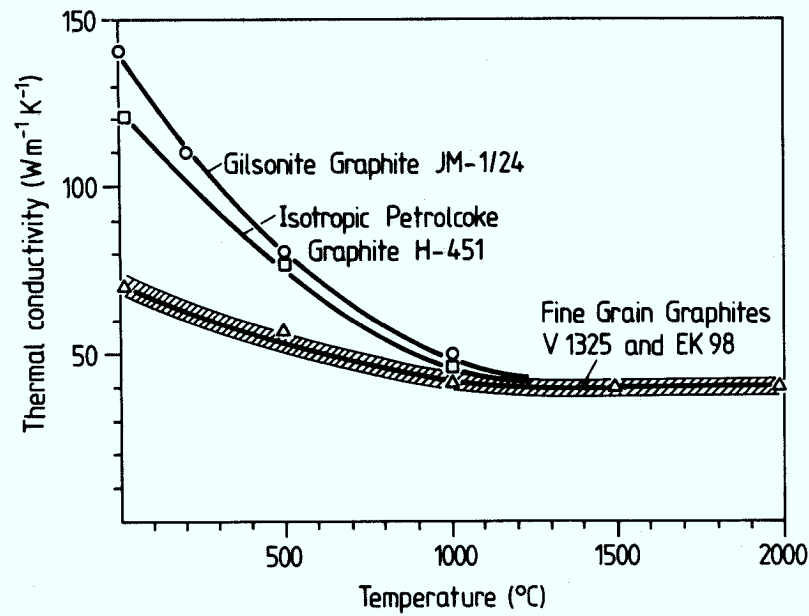


Fig. 3: Decrease of thermal conductivity with increasing measuring temperature for typical graphite qualities (Ref. 13, 16)

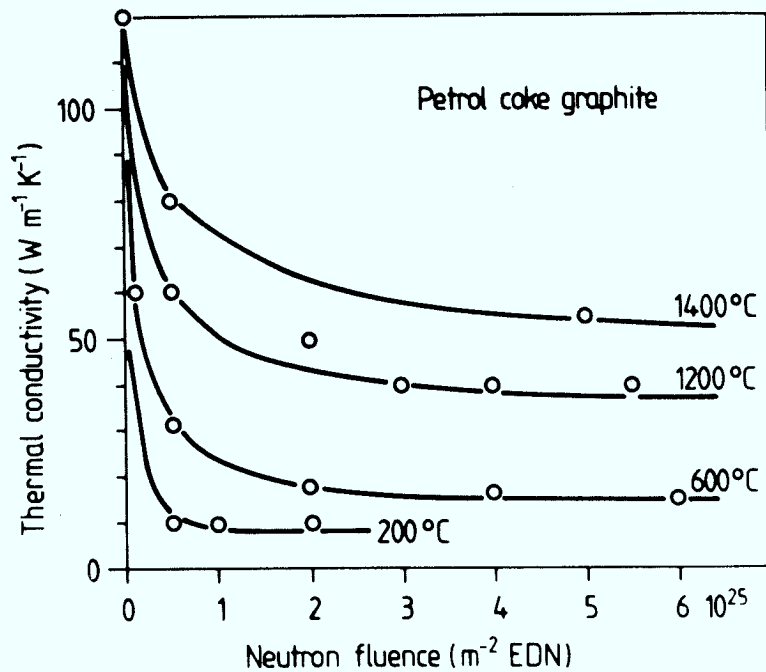


Fig. 4: Decrease of thermal conductivity of graphite by neutron irradiation for different irradiation temperatures; measuring temp. = room temp. (Ref. 13)

The coefficient of thermal expansion at room temperature varies for "conventional" nuclear grade graphites from about $3 \cdot 10^{-6} \text{ K}^{-1}$ - conventionally bonded fine grain graphite, e.g. EK 98 /16/ from Ringsdorff* - to about $8 \cdot 10^{-6} \text{ K}^{-1}$ - binder-free ultra-fine grain graphite, e.g. AXF 5Q /17/ from POCO** -.

The variation of the coefficient of thermal expansion as a function of temperature is not very pronounced /16/: If there is any at all, it will not exceed a factor of about 1,5, in most cases to higher values, between room temperature and 2000°C - again for the "conventional" qualities.

But on the other hand, the coefficient of thermal expansion varies - in some cases strongly - with the variation of the structural parameters and production parameters, such as:

- graphitizing temperature,
- type of filler and binder,
- binder content,
- grain size,
- density,
- factor of anisotropy of orientation.

Irradiation of graphitic materials with energetic neutrons has a strong effect /18,19/ on the coefficient of thermal expansion, fig. 5. In general one can say, that the coefficient is increased at the beginning of the irradiation, passes through a maximum, and then decreases with increasing neutron dose. The maximum is the higher the lower the irradiation temperature; the subsequent decrease is the stronger the higher the irradiation temperature.

* Ringsdorff-Werke GmbH, Bonn-Bad Godesberg, Fed. Rep. Germany

** POCO Graphite Inc., Decatur, Texas, USA

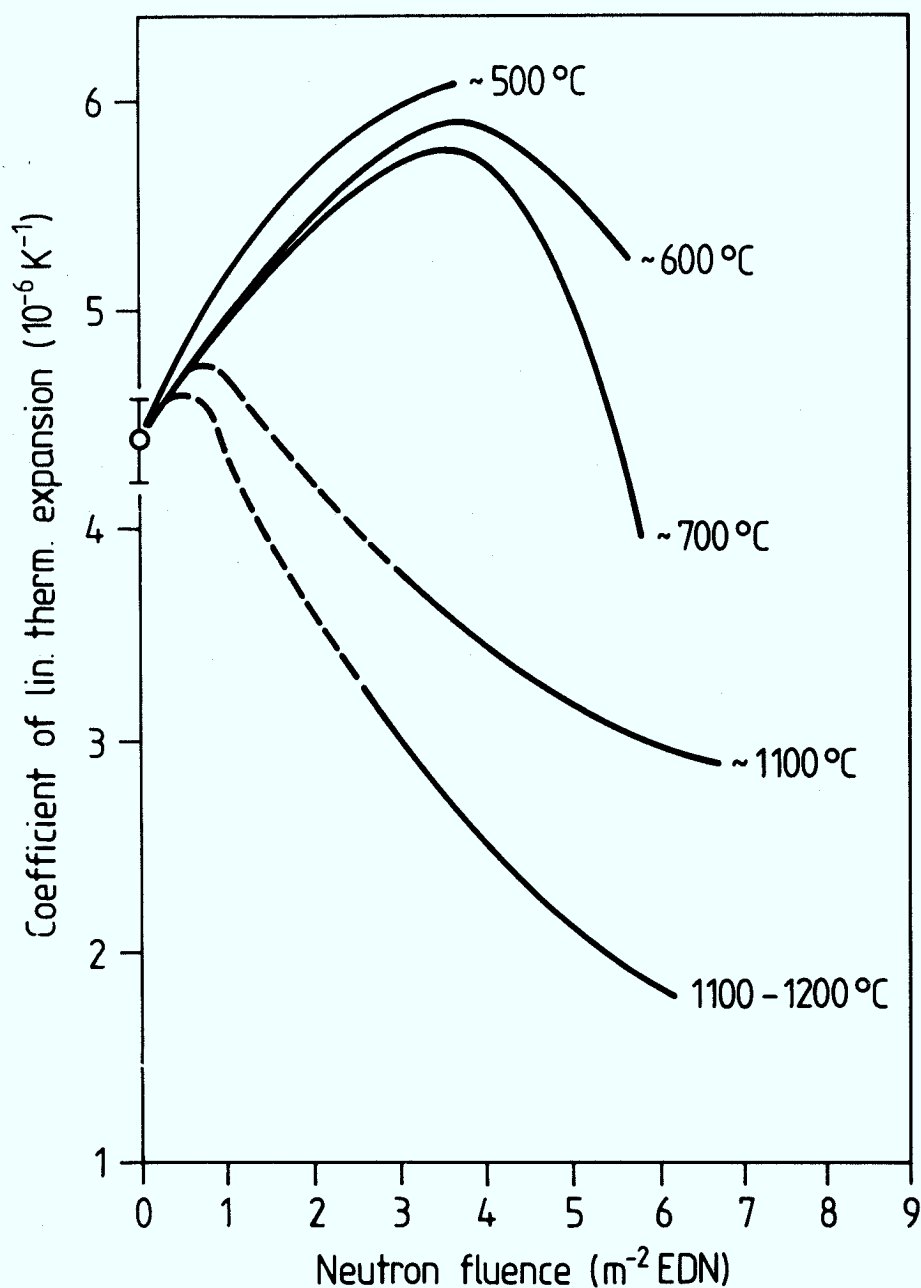


Fig. 5a: Coefficient of linear thermal expansion as a function of neutron fluence for different irradiation temperatures for the graphite type isostatically pressed petrol coke graphite 7477 (Ref. 18)

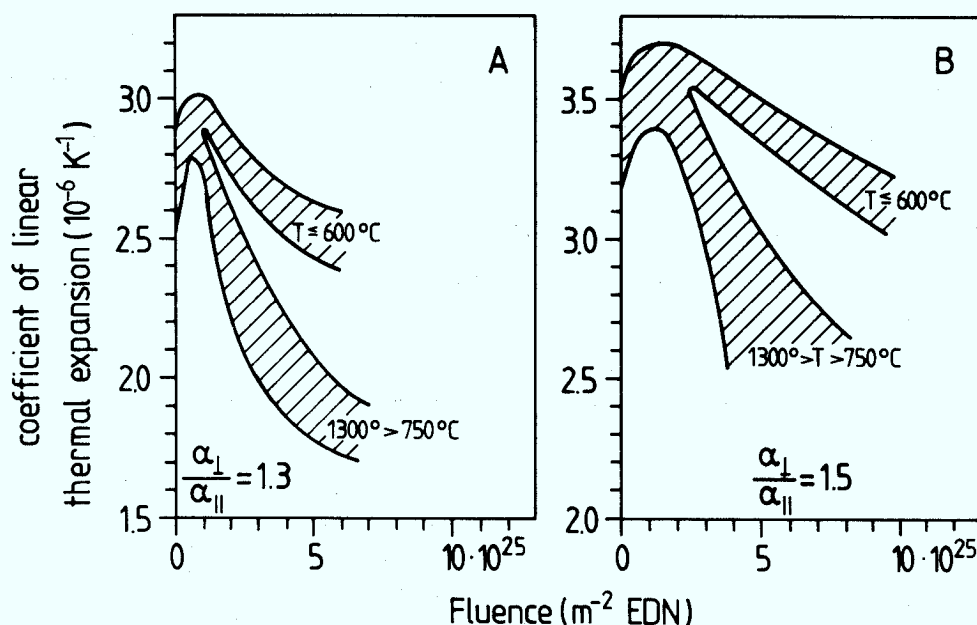


Fig. 5b: Coefficient of linear thermal expansion as a function of neutron fluence for different irradiation temperatures
A: petrol coke graphite
B: pitch coke graphite (Ref. 19)

3.1.3 Young's Modulus and Strength

Two important mechanical parameters are

- the modulus of elasticity or Young's modulus (E-modulus), and
- the strength.

The E-modulus, again, is strongly influenced by the production parameters and by other material characteristics. For "conventional" nuclear grade graphites its value is in the order of $10 \cdot 10^3 \text{ N mm}^{-2}$ at room temperature, for

the AXF-5Q /17/:	$11 \cdot 10^3 \text{ N mm}^{-2}$	= 11 GPa,
the EK-98 /16/:	$12 \cdot 10^3 \text{ N mm}^{-2}$	= 12 GPa,
the CL 5890 PT /20/:	$13,5 \cdot 10^3 \text{ N mm}^{-3}$	= 13,5 GPa *.

It is a function of temperature, and increases slowly with increasing temperature, although this increase is not very pronounced and may only reach a factor of 1.5 in the temperature range between 0°C and 2000°C.

* Le Carbone Lorraine, Gennevilliers, France

Neutron irradiation changes the value of the modulus of elasticity in that way that it is increased by the irradiation at low doses, and is decreased again at high doses. The "turn-around" of the E-modulus lies at about the same dose as the turn-around of the neutron induced dimensional changes; the neutron induced maximum is shifted to lower dose-values with increasing irradiation temperature /21/, fig. 6.

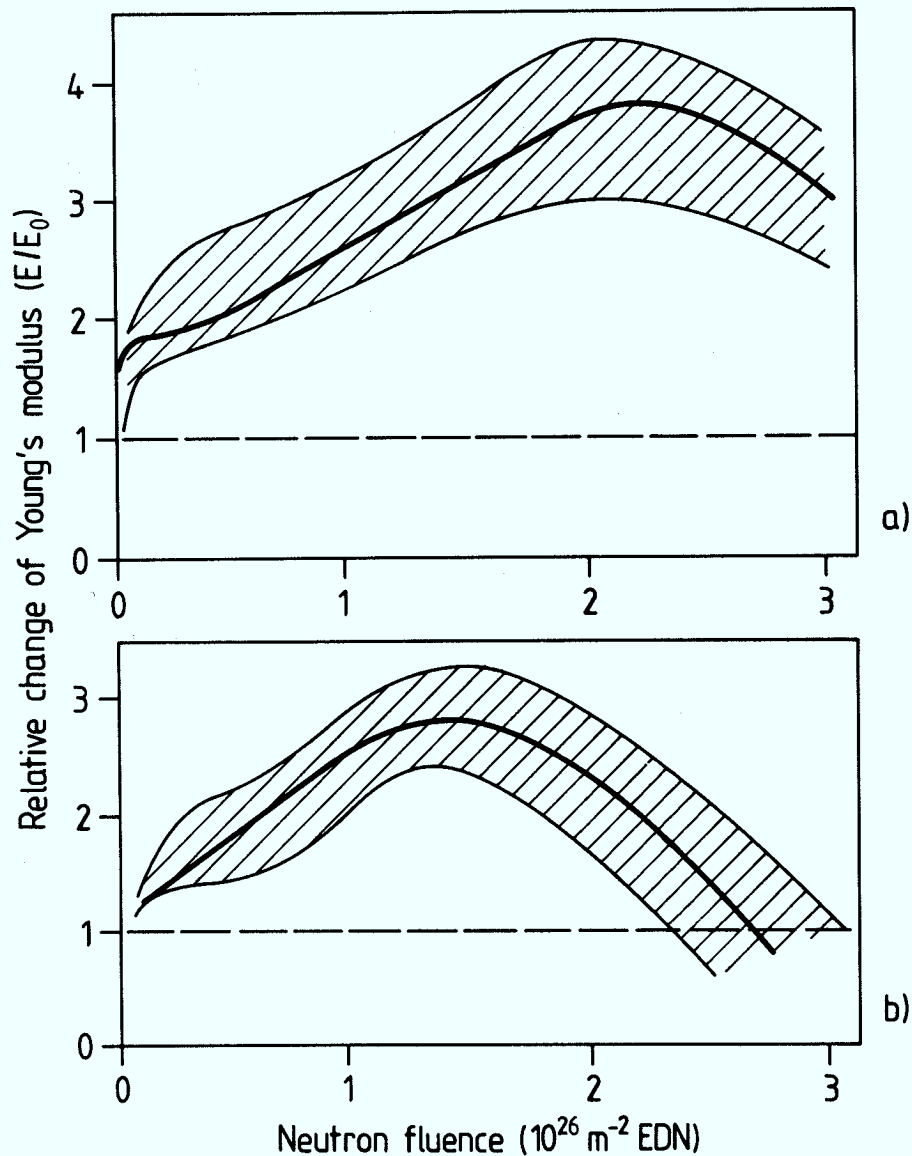


Fig. 6: Relative change of Young's modulus of different graphite qualities as a function of neutron fluence
a) irradiation temp. between 360 and 440°C
b) irradiation temp. between 560 and 790°C (Ref. 21)

In the case of the parameter "strength" one has to distinguish between three types of strength:

- the tensile strength, σ_T ,
- the compressive strength, σ_C ,
- the flexural strength, σ_F , or bending strength.

The tensile strength has proved to be a very conservative parameter /13/ for graphites, at least in the history of HTGR development. In the case of "non-exotic" graphites, one may assume compr. strength = $1.5 \cdots 2.5 \cdot$ flexural strength, and
flex. strength = $1.5 \cdots 2.5 \cdot$ tensile strength.

So it would make sense to use the flexural strength, e.g. in the form of the three-point- or four-point-bending-strength, as a characterization parameter for graphites, but data published so far mention mostly non-systematically one or two of the three parameters, and in some cases all the three.

At room temperature, the values of strength for "conventional" nuclear grade graphite are, for example:

AXF-5Q: $\sigma_F = 90 \text{ N mm}^{-2}$

$\sigma_C = 155 \text{ N mm}^{-2}$

$\sigma_T = 62 \text{ N mm}^{-2}$

EK-98: $\sigma_F = 50 \text{ N mm}^{-2}$

$\sigma_C = 125 \text{ N mm}^{-2}$

CL-5890-PT: $\sigma_T = 50 \text{ N mm}^{-2}$

So one can say that at room temperature the values for the "conventional" graphites are in the range of: for the
tensile strength σ_T : 15 N mm^{-2} to 60 N mm^{-2} ,
flexural strength σ_F : 25 N mm^{-2} to 100 N mm^{-2} .
compr. strength σ_C : 80 N mm^{-2} to over 160 N mm^{-2} .

As can be deduced from these wide scatter bands, the values of the strength depend strongly on the other material parameters and production data, and they are a function of measuring temperature /22/, in particular at elevated temperatures, fig. 7.

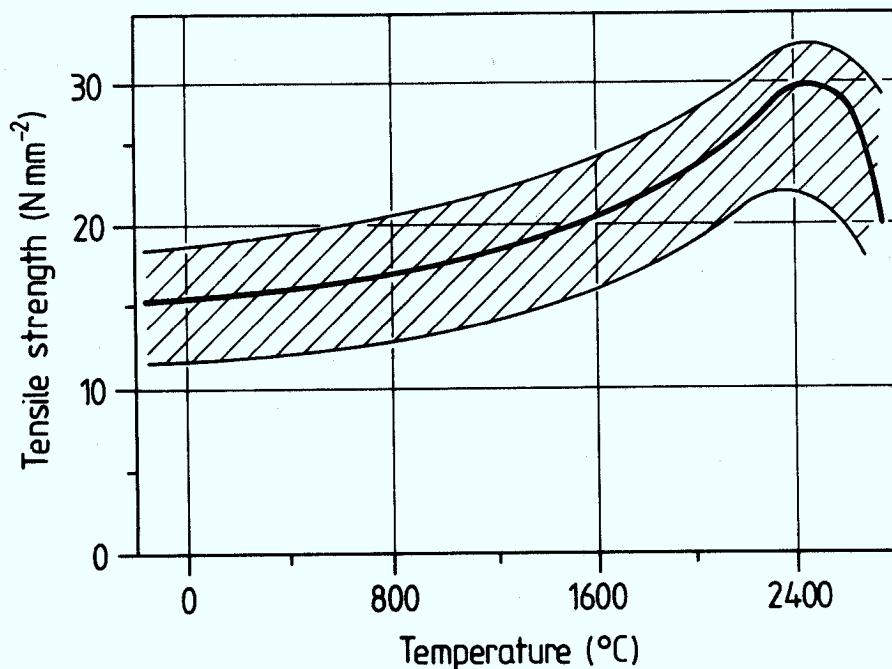


Fig. 7: Tensile strength of the petrol coke graphite ATJ as a function of measuring temperature (Ref. 22)

Of course, the strength is also affected by neutron irradiation. As can be seen in fig. 8 for the tensile strength /23/, the effect is not very pronounced: The tensile strength slowly increases for many graphite qualities, or remains more or less constant. There is also no clear dependence of the tensile strength on the irradiation temperature. The only slight or even negligible effect of neutron irradiation and irradiation temperature on the strength of graphitic material is surprising if one keeps in mind the strong influence on the parameters

- thermal conductivity,
- thermal expansion, and
- modulus of elasticity, and finally, what shall be discussed now,
- dimensional changes.

3.1.4 Dimensional Changes

The neutron induced dimensional changes are the most extensively investigated effects on graphites. The behaviour of the dimensional changes - again for "conventional" nuclear grade

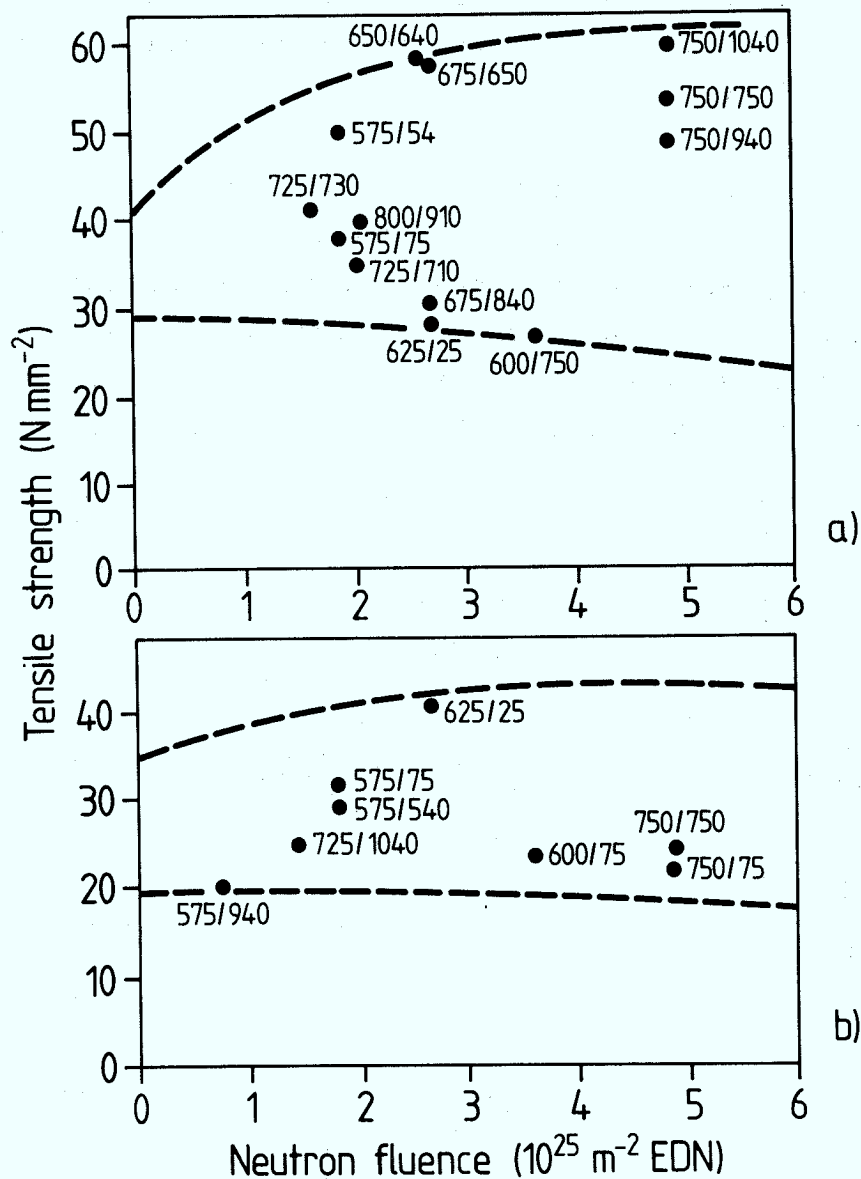


Fig. 8: Tensile strength as a function of neutron fluence for different graphite qualities and for different irradiation temperatures; figures at the measuring points: irradiation and measuring temperatures; resp.; all samples parallel grain orientation; a) samples taken from the outer zones of the graphite blocks, b) samples taken from the center parts of the graphite blocks (Ref. 23)

graphites - is principally similar for all materials and shows a strong dependence on neutron dose and irradiation temperature, fig. 9. Fig. 10 /13/ shows the dimensional changes for a particular quality.

In general one can say that the graphite shrinks at the beginning of the irradiation, passes through a "turn-around", expands again, passes through the original dimensions, and finally begins a net expansion. The turn-around is reached at lower neutron doses when the irradiation temperature is higher; for many graphite qualities is the absolute value of the neutron

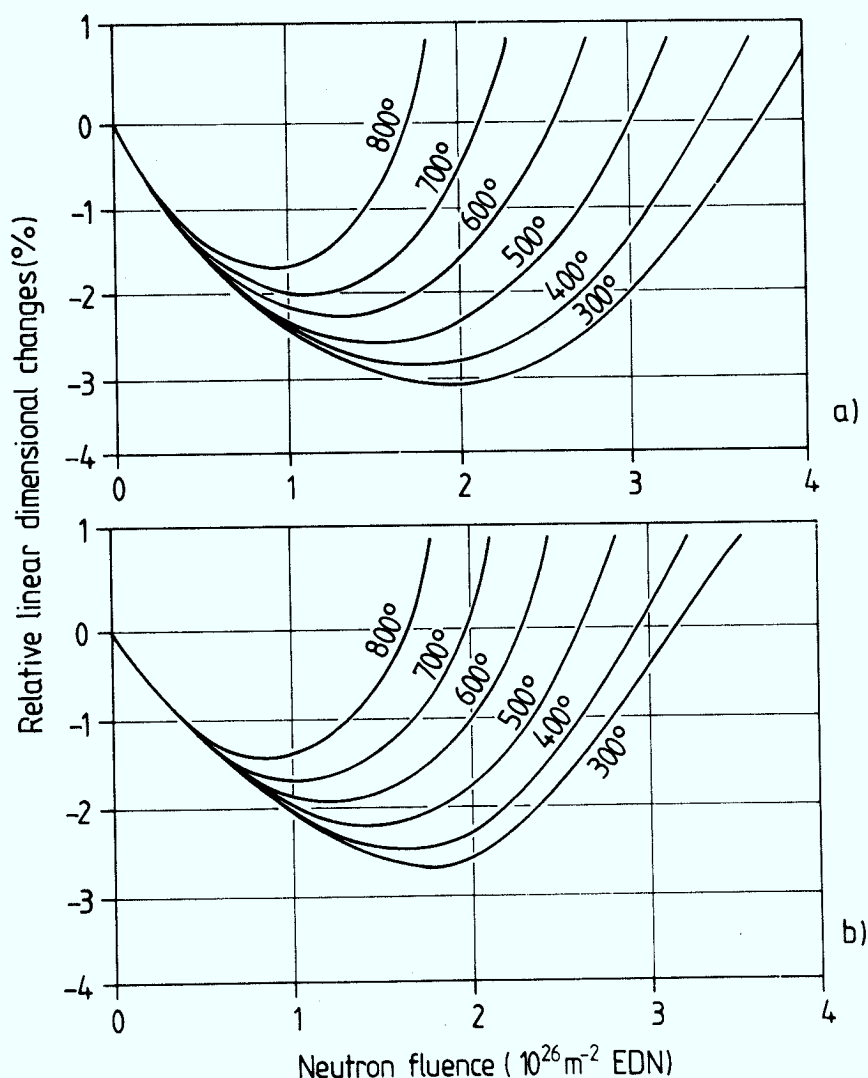


Fig. 9: Relative linear dimensional changes as a function of neutron fluence, for different irradiation temperatures, for nearly isotropic pitch coke graphite
a) parallel to grain orientation
b) vertical to grain orientation (Ref. 13)

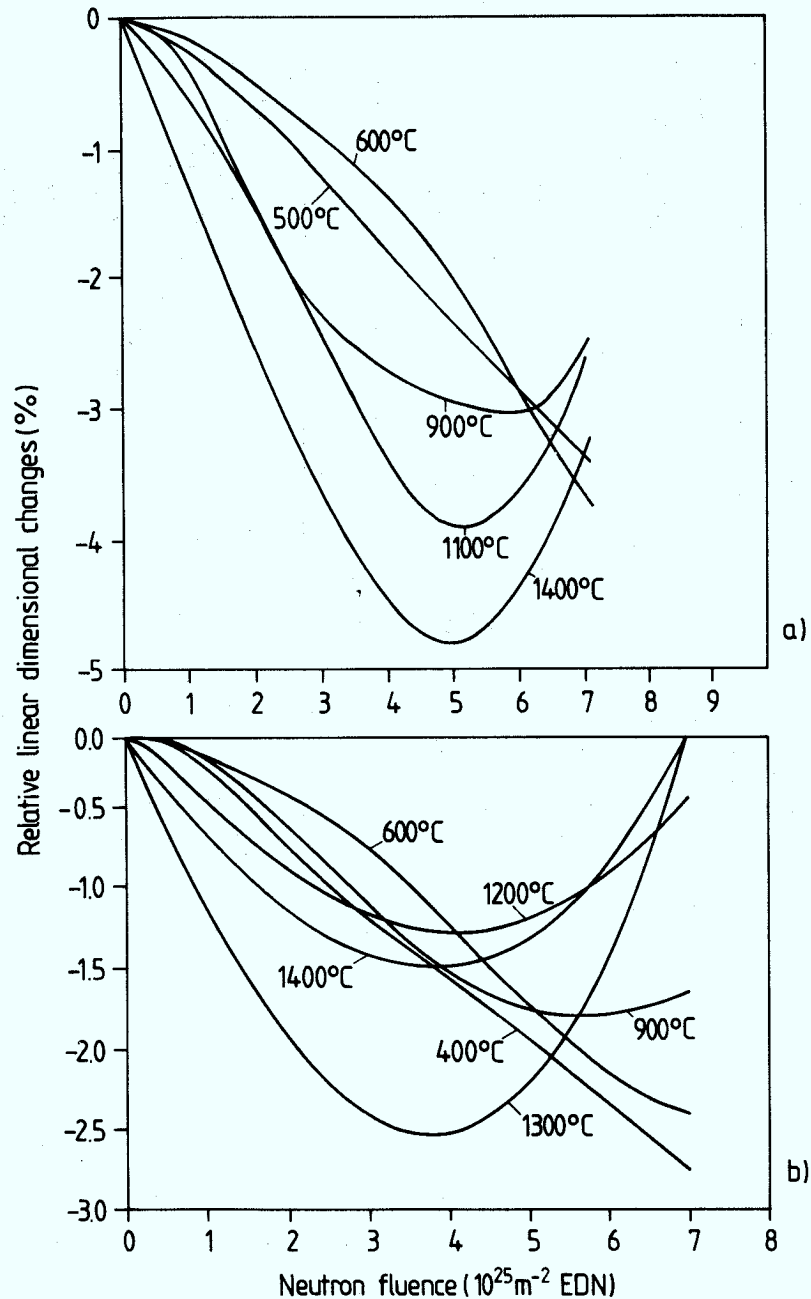


Fig. 10: Relativ linear dimensional changes as a function of neutron fluence, for different irradiation temperatures, for the graphite quality petrol coke graphite ASR-2E,
a) parallel to grain orientation
b) vertical to grain orientation (Ref. 13)

induced shrinkage at the turn-around the higher the higher the irradiation temperature.

The strong dependence of the neutron induced dimensional changes on the material and production parameters shall be exemplified by the influence of the degree of graphitization on the dimensional changes /18/, fig. 11.

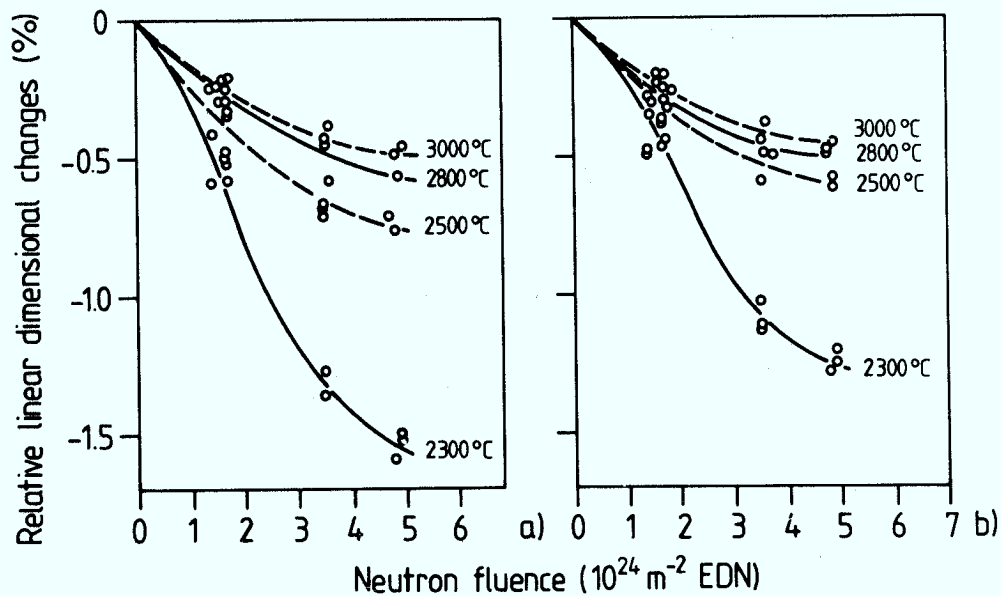


Fig. 11. Relative linear dimensional changes as a function of neutron fluence for graphitic materials which differ only in their graphitizing temperature; irradiation temperature between 1060 and 1280°C; figures at the measuring curves: graphitizing temperatures;
a) parallel to grain orientation
b) vertical to grain orientation (Ref. 18)

The conclusion may be drawn from fig. 11 that the dimensional changes decrease with increasing graphitizing temperature. But one has to note that the anisotropy of orientation may also increase with increasing degree of graphitization and by this effect the anisotropy of dimensional changes, fig. 12 /13/.

This interrelation shall only be an example for the very complex, mutual dependences that are operative between the different material and production parameters and their neutron induced changes.

3.1.5 Other Parameters

There are, of course, more parameters which are important for the valuation of a material with respect to its applicability

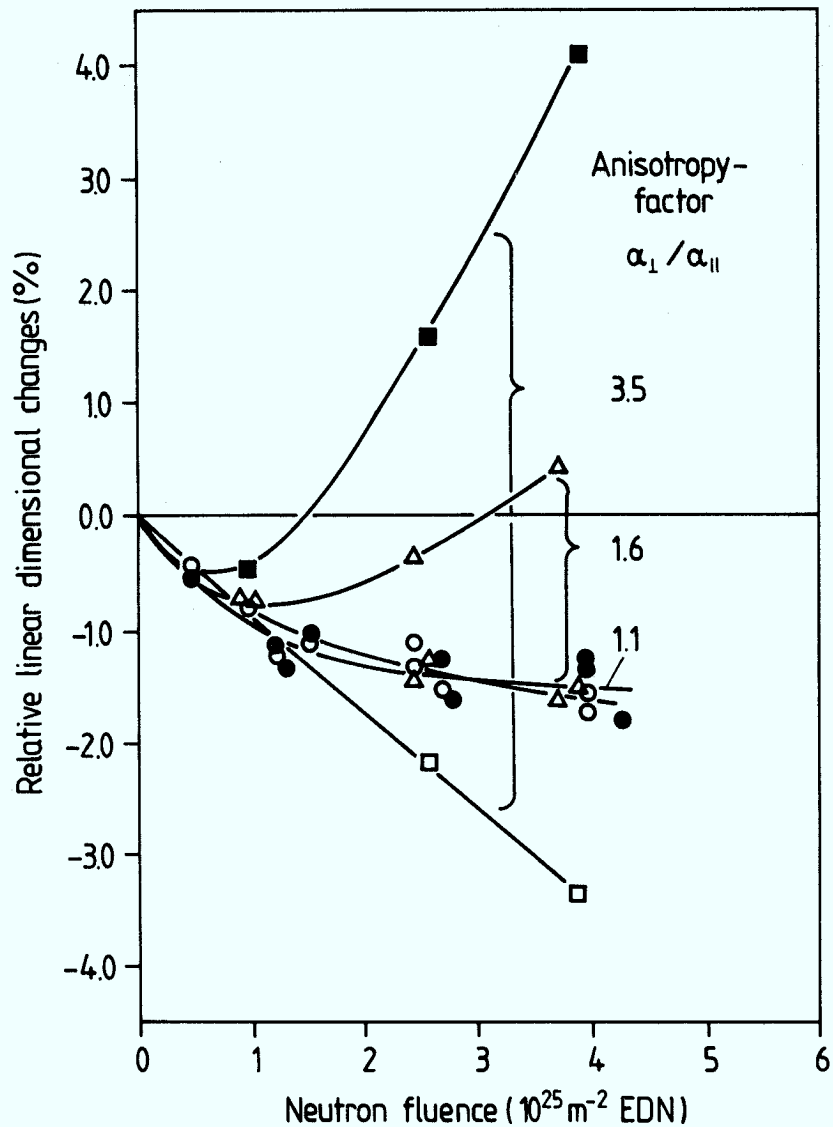


Fig. 12: Relative linear dimensional changes of graphitic matrix materials differing only in their anisotropy of orientation, as a function of neutron fluence; the anisotropy of orientation is given as the ratio of the coefficients of thermal expansion vertical ($\alpha_{\perp}$) and parallel ($\alpha_{\parallel}$) to the grain orientation (Ref. 13)

in the first wall of a fusion device, like

- creep constant,
- porosity,
- hydrogen/tritium retention and diffusion, resp.
- fatigue behaviour under cyclic load, and may be some more.

But it would exceed the intention of this report to discuss also these parameters.

Only general remarks can thus be made concerning the behaviour of a material under varying flux of thermal energy and neutron irradiation. This fact makes it necessary to investigate finally the behaviour of each particular first wall candidate material.

3.2 Other Materials

It shall only be pointed out that data on the irradiation behaviour of three other material classes exist which include additional candidates for first wall components, namely

- carbon fiber reinforced carbons (CC's),
- highly oriented, highly graphitized graphites (pyrographites and pyrocarbons), and
- silicon carbides.

Only relatively few data are available so far for the first two material classes. Their systematic investigation with respect to fusion application has started just recently. One example for the behaviour of CC's under neutron irradiation /24/ is given in fig. 13, from which one can deduce that, compared to

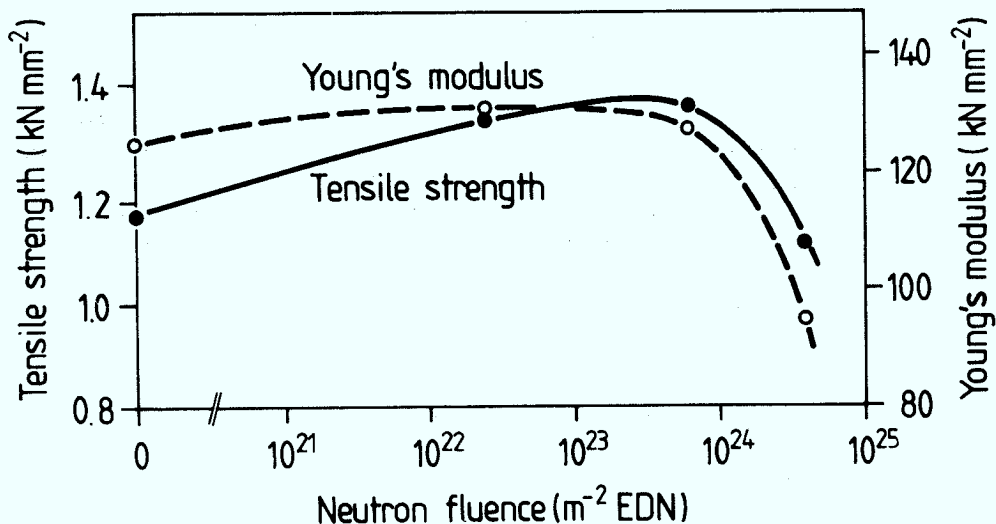


Fig. 13: Tensile strength and Young's modulus as a function of neutron fluence for a CC-material; irradiation temperature about 200°C (Ref. 24)

graphites, CC's have much higher values of the modulus of elasticity (by a factor of about 10) and of the tensile strength (by a factor of about 20), both in fiber direction; but the fast, neutron induced deterioration of these mechanical properties begins already at very low dose values, in the example shown here already at a dose of about 10^{24} cm^{-2} .

In the case of pyrographite, the neutron induced dimensional changes are fairly well known. Here, the anisotropy plays an important role, as has already been pointed out.

The situation in the case of silicon carbide materials is not much better. Only few data exist so far for this material class. From these few informations a first conclusion might be drawn in that sense that the thermal conductivity, for example, behaves similar under neutron irradiation as in the case of graphites and drops considerably at low irradiation temperatures already at medium neutron fluences /25/, fig. 14, i.e. at neutron fluences about 10^{25} m^{-2} .

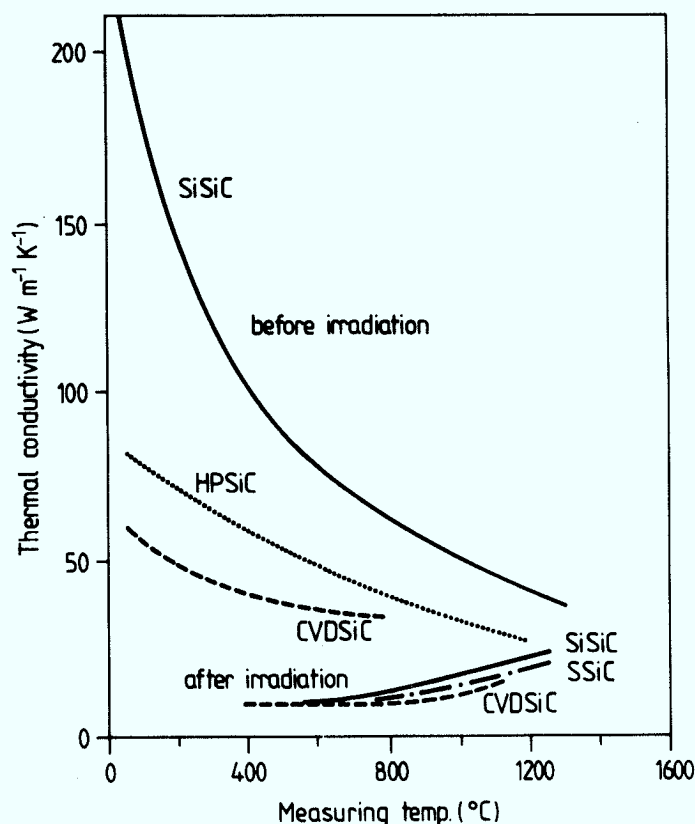


Fig. 14: Decrease of thermal conductivity of different qualities of silicon carbide by neutron irradiation with a fluence of 10^{25} to 10^{26} m^{-2} , as a function of measuring temperature (Ref. 25)

4. TEST PHILOSOPHY AND AVAILABLE NEUTRON SOURCES

The data and results presented in chapter 3 allow two general conclusions, one concerning the critical neutron doses and irradiation temperatures for the respective material properties, the other concerning the relevance of the available neutron sources. And, of course, there is a third open question that has to be looked at, namely the investigation of the irradiation behaviour of the actual candidate materials, since the available data have mostly been measured for "similar" materials or for a "typical" graphite but not for the actual candidate.

4.1. Irradiation Parameters and Material Properties

Running through the discussed neutron effects, primarily in graphite, one can define some typical "problem areas" basing on the irradiation parameters and concerning the effects of the energetic neutrons on the material properties. These "problem areas" have to be investigated in more detail with respect to fusion technology.

The first critical area is the thermal conductivity and its decrease by neutron irradiation. The thermal conductivity is decreased extremely rapidly at the beginning of a neutron irradiation, that means, already at very low fluences, i.e. at fluences below $0.5 \cdot 10^{25} \text{ m}^{-2} \text{ EDN}$.

This effect is the smaller the higher the irradiation temperature, and one may assume that at irradiation temperatures above 1500°C the effect of the energetic neutrons on the thermal conductivity will play no decisive role.

Since thermal conductivity is one of the most important characteristics for the plasma facing material this effect has to be investigated carefully, and this has to be done for each candidate material or at least for each group of candidate materials.

A group of irradiation experiments result from this fact for graphites and other carbon-based materials: To investigate the rapid decrease of thermal conductivity irradiation experiments have to be carried out at low and medium temperatures and at low neutron fluences. One irradiation test might be useful at a high temperature - about or even above 1500°C - and with a neutron fluence about $0.5 \cdot 10^{25} \text{ m}^{-2}$ to prove that the assumption of no considerable decrease of thermal conductivity under these conditions is really valid.

The second "problem area" concerns the thermal expansion of graphitic materials. The energetic neutrons affect the coefficient of thermal expansion primarily under the following conditions:

- irradiation temperature above 1000°C , neutron fluence about or above $2 \cdot 10^{25} \text{ m}^{-2}$: strong decrease of the coefficient of thermal expansion, and
- irradiation temperature about 600°C , neutron fluence again about or above $2 \cdot 10^{25} \text{ m}^{-2}$: strong increase of the coefficient of thermal expansion with a subsequent rapid decrease.

Both effects can cause severe problems for first wall components when bonded structures are used as plasma facing materials. Thus they have to be investigated in detail. This is the reason for another two groups of irradiation experiments, both at medium neutron fluences of 2 to $5 \cdot 10^{25} \text{ m}^{-2}$ and at irradiation temperatures of about 1300 to 1500°C and of about 400 to 600°C , resp.

The third parameter and its variation by neutron irradiation which has to be looked at is the Young's modulus. Here, one can discriminate two fluence bands in which important effects occur, namely very low fluences, clearly below $1 \cdot 10^{25} \text{ m}^{-2}$, by which a rapid increase of the modulus is caused, and fluences of about $10 \cdot 10^{25} \text{ m}^{-2}$ and higher, at which the increase of the Young's modulus turns around to a re-decrease.

The Young's modulus is one of the parameters which obviously determine quantitatively the "thermal shock coefficient" or "figure of merit" given by the relation

$$G = \frac{\sigma_B \cdot \lambda}{E \cdot \alpha}$$

where σ_B = fracture strength,

λ = thermal conductivity,

E = Young's modulus,

α = coefficient of thermal expansion.

So it is necessary to investigate the irradiation behaviour of the Young's modulus, and this suggests two irradiation experiments, one at temperatures about 500°C and neutron fluences about $0.5 \cdot 10^{25} \text{ m}^{-2}$, the other at about the same temperature and neutron fluences of about $20 \cdot 10^{25} \text{ m}^{-2}$.

A third experiment might make sense at a temperature of about 1000°C or even higher and a neutron fluence about or below $1 \cdot 10^{25} \text{ m}^{-2}$ to look whether the shift of the turn-around to lower fluence values at higher irradiation temperatures has really to be taken into consideration.

The strength and its variation by neutron irradiation, on the other hand, could be neglected at a first approach because of the obviously only small effects of the energetic neutrons. If at all, one should examine the behaviour of the strength under neutron irradiation in an experiment at very high neutron doses, e.g. above $50 \cdot 10^{25} \text{ m}^{-2}$.

In the case of SiC, the neutron induced decrease of the flexural strength is more pronounced /26/ than for graphite, obviously strongly depending on the type of the material, fig. 15.

Concerning neutron dose and irradiation temperature, there seem to be no particular dependences except that the flexural strength decreases with increasing fluence and that a strong influence of the quality of the material can be stated. Some systematic irradiation experiments, particularly to higher neutron fluences should yield some more general informations.

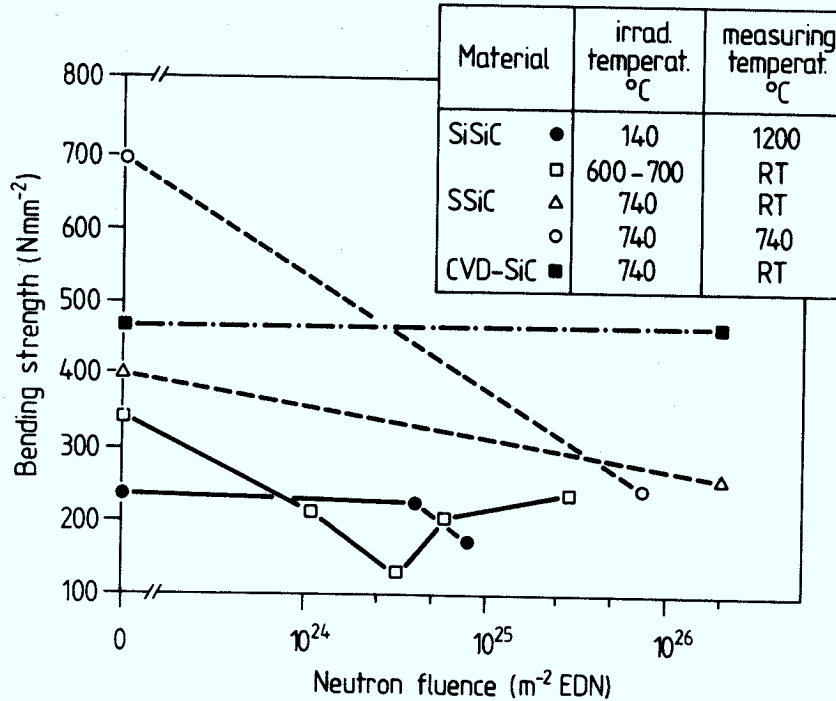


Fig. 15: Bending strength as a function of neutron fluence for different types of silicon carbide; material qualities, irradiation temperatures, and measuring temperatures are given in the inserted table (Ref. 26)

Particular problems exist for CC materials. Here, a very rapid degradation of Young's modulus and strength is caused by the energetic neutrons already at very low fluences, at least as far as the only few experimental results of irradiation tests from the past allow this general conclusion. So, irradiation experiments with CC materials under different aspects are urgently necessary.

Finally, the dimensional changes by energetic neutrons have to be mentioned. Disregarding the strong influence of the material and production parameters they show a relatively uniform behaviour, at least for "conventional" nuclear grade graphite: The dimensional changes are the more pronounced the lower the irradiation temperature; the turn-around is reached at lower neutron doses and the subsequent expansion is more rapid when the irradiation temperature is higher.

There is no irradiation temperature at which dimensional changes could be neglected, and the most important ranges of the neutron

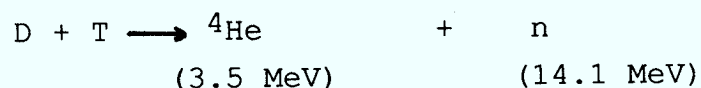
fluence are 2 to $5 \cdot 10^{25} \text{ m}^{-2}$ for the shrinkage of the material, and about $10 \cdot 10^{25} \text{ m}^{-2}$ for the expansion.

As for itself, the dimensional changes seem to be not very important for graphitic materials for fusion application, at least at low fluences - below $2 \cdot 10^{25} \text{ m}^{-2}$ - and at elevated temperatures. They have to be taken into account in the case of bonded structures - e.g. graphite as the plasma facing material, brazed to Mo or some other high-Z metal -, at lower temperatures and higher neutron fluences, and in particular they have to be investigated for the anisotropic pyrographites, pyrocarbons, and for the CC materials.

From these considerations one can draw the following conclusion: As far as "conventional" graphites are irradiated at temperatures and neutron fluences at which dimensional changes occur at all, these changes should be measured. But special irradiation experiments for that purpose for bare, that means for unbonded graphite seem to be unnecessary at the moment. This is no longer true for pyrographites, pyrocarbons, and CC: Here, special irradiation experiments will have to be carried out for some typical first wall temperatures and for characteristic neutron fluences to get at least first systematic and reliable informations on the material behaviour.

4.2 Available Neutron Sources

The D-T-fusion reaction produces highly energetic neutrons according to the equation



During their lifetime, the neutrons become moderated. Thus the energy spectrum of the neutrons in a fusion device operating on the D-T-process will cover the full range from 14 MeV to thermal energy, i.e. 0.025 eV. It can be assumed that the shape of the energy distribution will be more or less similar to that of a

fast breeder fission reactor, that means that it will show some type of maxima at and just below 14 MeV and at and just above thermal energy, and between both maxima a more or less flat path. The actual shape, of course, will depend on the first wall materials and the geometry of the first wall components, and it will probably show a local variation with respect to the geometry of the plasma and the fusion reactor. A calculated energy spectrum /27/ in the graphitic first wall of a fusion reactor for neutron energies above 10 eV is given in fig. 16.

Up to now no neutron source exist with a realistic neutron energy spectrum and a sufficiently high neutron flux for material tests. Two types of neutron sources are available: spallation neutron sources with upper energy limits - depending on the resp. spallation process - up to several 100 MeV, and fission reactors with upper energy limits of about 2 MeV.

In the case of fission reactors again two different types of this device are in principle available, namely material test reactors and fast breeder reactors. The neutron spectrum of fast breeder reactors is - under the aspect of fusion material tests - "harder" than that of material test reactors, but with no basic difference concerning the material - neutron interaction.

Concerning only the neutron induced lattice defects in the non-metallic solids regarded here the energetic fission neutrons and the fusion neutrons will not differ in their effects, i.e. in the type of displacements they produce, but in the number of produced displacements per atom for a certain neutron dose. At a first approach one can calculate /28,29,30/
for graphite: $1 \cdot 10^{25} \text{ m}^{-2} \text{ (E > 0.1 MeV)} = 1 \text{ dpa}$
for SiC: $0.5 \cdot 10^{25} \text{ m}^{-2} \text{ (E > 0.1 MeV)} = 1 \text{ dpa}.$

The damage rate for 14 MeV - fusion neutrons is calculated approximately
for graphite: $4 \cdot 10^{25} \text{ m}^{-2} \text{ (E} \sim 14 \text{ MeV)} = 1 \text{ dpa}.$

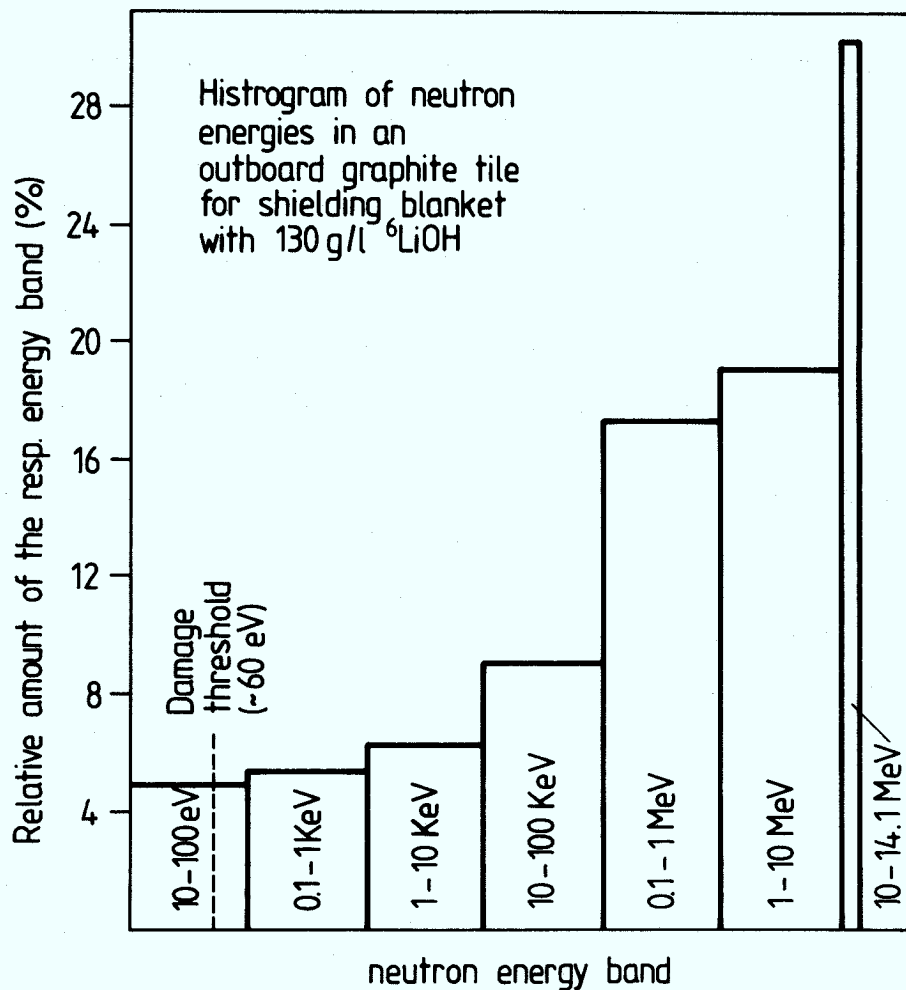


Fig. 16: Possible shape of the neutron energy spectrum in a fusion reactor for energies above 10 eV, i.e. for the epi-thermal and energetic part of the neutron spectrum (Ref. 27)

The damage rate for a realistic fusion neutron spectrum is presently expected to be

for graphite: from $1 \cdot 10^{25} \text{ m}^{-2} = 1 \text{ dpa}$

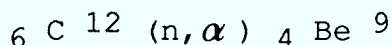
to $5.5 \cdot 10^{25} \text{ m}^{-2} = 1 \text{ dpa}$,

with the latter value, about $5.5 \cdot 10^{25} \text{ m}^{-2}$ corresp. to 1 dpa, being presently considered to be the most likely one.

So the conclusion seems to be valid that a numerical uncertainty exists for the conversion of different neutron spectra to fusion reactor conditions with respect to neutron induced lattice defects. But nevertheless it is undoubtedly useful to perform irradiation experiments in fission reactors to study the basic

damage processes and to get at least "semi-quantitative" informations. In the case of spallation neutron sources the situation is more complicated because of the possibly extremely high neutron energy. But again one can say that at least few experiments in well known spallation spectra can be useful to produce some basic informations about the irradiation behaviour of solids.

Irradiation experiments in fission reactors can no longer be useful when transmutation effects shall be studied. In particular in carbon the important transmutation



by which He is produced starts at neutron energies significantly higher than the maximum fission neutron energy of 2 MeV, possibly at energies above 6 MeV /11/. On the other hand, the neutron energies in spallation sources are high enough, but the available neutron fluxes are very low, and the effects of the very high neutron energies are not yet known. Irradiation experiments in the available neutron sources are thus primarily restricted to lattice damage effects, and should be carried out in suitable fission reactors, except from very few materials tests in spallation neutron sources. This will be true as long as no realistic fusion neutron source is available for materials tests.

5. IRRADIATION TESTS AND MATERIALS SELECTION

Following the considerations discussed in chap. 4 the Institute for Reactor Materials has planned and is performing a first group of irradiation experiments - partially in cooperation with two other research centers*, and in accordance with the fusion project of KFA** and the NET Team***, which is respon-

* Nuclear Research Center, Karlsruhe, Fed. Rep. Germany;
Centre d'Etude Nucleaire de Saclay, Gif-Sur-Yvette, Saclay, France;

** Project Plasma- and Wall-Technology PWT, Nuclear Research Center, Jülich; Fed. Rep. Germany;

*** The NET Team, Max-Planck-Inst. f. Plasmaphysics, Garching, Fed. Rep. Germany

sible for the design and development of the NET device and is decisively jointly responsible for the design of the ITER. The materials tested in these irradiation experiments are non-metallic first wall candidates which have been or are taken into consideration as plasma facing materials. They belong to the two groups "carbon based materials" and "thermally conductive ceramics".

5.1 Irradiation Tests

The IRW participates in or performs at present six irradiation experiments, five of them in fission reactors, one in a spallation neutron source, fig. 17.

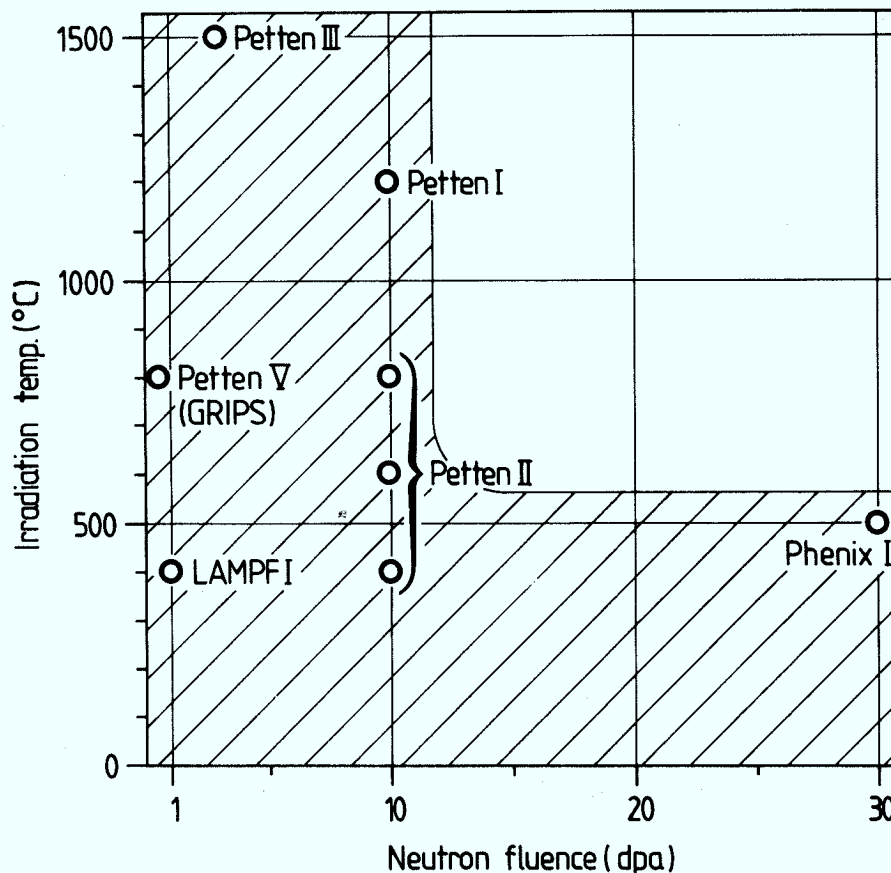


Fig. 17: The fusion-oriented irradiation experiments of IRW, performed or prepared up today; irradiation temperature and neutron fluence give nominal values for the resp. experiment

The names of the irradiation experiments given in fig. 17 refer to the resp. neutron source:

"Petten"- experiments are carried out in the High Flux Materials Testing Reactor HFR /31/ at the Petten Joint Research Center (JRC) of the European Communities in Petten, The Netherlands. This reactor is cooled and moderated by light water, is operated with 93 % enriched Uranium and is working at nominal 45 MW and irradiation temperatures below 1500°C.

"LAMPF" is an experiment in the Los Alamos Meson Physics Facility /32/, a spallation facility that operates with highly energetic proton beams up to 800 MeV on metal targets of medium or high Z. This facility provides neutrons with a maximum flux of $6 \cdot 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$ and at energies up to 100 MeV. The maximum irradiation temperature is about 650°C.

"Phenix" refers to the French fast breeder prototype reactor in Marcoule.

The irradiation experiment Petten V (GRIPS) summarizes a group of low-dose experiments to investigate the decrease of thermal conductivity already at low neutron doses, fig. 18. The one sub-group which is under preparation already and shall start in 1989 in that one at a nominal irradiation temperature of 800°C.

The irradiation experiments Petten III and Petten V are carried out in the frame of technology contracts in the European Fusion Technology Program.

5.2 Materials Selection and Characterization

The materials selected for the mentioned irradiation experiments belong primarily to the three materials groups

- fine grain and ultra-fine-grain graphites,
- thermal conducting ceramics, and
- carbon fiber composites.

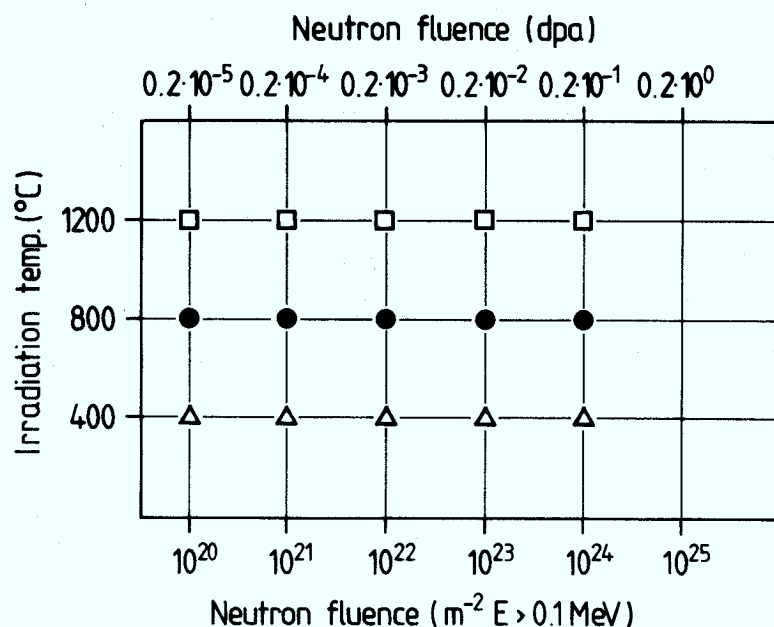


Fig. 18: The Petten V-(GRIPS) irradiation experiment is a "family" of three "sub-groups" of low fluence irradiation test at low, medium and high irradiation temperature to investigate the neutron induced decrease of thermal conductivity of graphites and other carbon based materials.

Two more "exotic" materials are included into two experiments, namely a silicon carbide fiber reinforced silicon carbide, SiC-SiC, and a quality of oriented pyrographite. The complete loading is given in tab. 1, the sample geometries and dimensions for each experiment in tab. 2.

The nominal material properties, mainly as being published by the producers /16,17,20,33-39/, are listed in tab. 3. A more detailed characterization of the materials will be given in the resp. pre-irradiation reports, which are to be published shortly.

The main material characteristics to be investigated are

- thermal conductivity,
- thermal expansion,
- neutron induced dimensional changes,
- Young's modulus,
- bending strength and
- thermal shock behaviour.

		Petten I responsible KfK	Petten II	Petten III split into 2 parts; part 1 for SiC (res. KfK)	Petten V GRIPS	LAMPF I	Phénix I responsible Saclay
EK 98	Ri		x	x	x	x	
FP 219	Sch					x	
5890 PT	CL			x	x	x	
S 1611	CL			x	x		
S 1260	CL		x	x		x	
V 1325	Ri			x	x		x
ASR-1 RS	Si	x					
BN	ESK		x			x	x
Si ₃ N ₄	ESK		x			x	x
SiC + 5% AlN	ESK		x	x separate experim.		x	x
SiC + 2% AlN	Hi		x only 800 C			x	
SiC + 2% AlN	ESK	x					
SiC + 2% BeO	Hi		x only 800 C			x	
Aerolor 05	CL			x	x		
Aerolor 46	CL			x			
FMI 4D 333-6	FMI			x	x		
CF 222	Sch			x	x		
SiC-SiC Oriented Pyrographite	GA Fi				x	x	

Tab. 1: Materials Loadings of the irradiation experiments
(or parts of the experiments) of IRW

Producers:

Ri: Ringsdorff-Werke, Bonn-Bad Godesberg, Fed. Rep. Germany
 Sch: Schunk-Kohlenstoff, Gießen, Fed. Rep. Germany
 CL: Le Carbone Lorraine, Gennevilliers, France
 Si: Sigri Elektrographit, Meitingen, Fed. Rep. Germany
 ESK: Elektroschmelzwerk, Kempten, Fed. Rep. Germany
 Hi: Hitachi, Tokyo, Japan
 FMI: FMI Composites, Galashiels, Scotland
 GA: General Atomics, San Diego, USA
 Fi: Pfizer Inc., Easton, USA

	Type of test and specimens	size/mm
Petten I	Bending test (SiC + 2% AlN) Thermal shock test (e ⁻ -beam) (SiC + 2% AlN) Swelling/therm. expansion (graphite)	3.5 × 4.5 × 45 13 Ø × 10 6 Ø × 25
Petten II	Bending test (BN, Si ₃ N ₄ , SiC + 5% AlN) Thermal and temperature conductivity (BN, Si ₃ N ₄ , SiC + 5% AlN) Reference material (graphite)	3.5 × 4.5 × 45 25 Ø × 28 or 12.7 Ø × 4 6 Ø × 32 and 6 Ø × 25
Petten III	Bending test (SiC + 5% AlN) Thermal shock test (e ⁻ -beam) (graphite, CC) Thermal conductivity (graphite, CC) Tritium retention (graphite, CC)	3.5 × 4.5 × 45 22 Ø × 9 6 Ø × 25 or 6 Ø × 32 22 Ø × 1
Petten V	Thermal conductivity (graphite, CC)	6 Ø × 32
LAMPF I	Bending test (all ceramics) Thermal conductivity (all graphites) Thermal conductivity (all ceramics) Thermal diffusivity (all ceramics)	3.5 × 4.5 × 45 6 Ø × 25 or 6 Ø × 32 25 Ø × 28 10 Ø × 4 or 12.7 × 4
Phénix I	Bending test (all ceramics) Thermal conductivity (V 1325)	3.5 × 4.5 × 45 6 Ø × 25

Tab. 2: Types of tests (PIE) and specimens, and size of samples for the present IRW irradiation experiments; the materials samples from other partners are not listed.

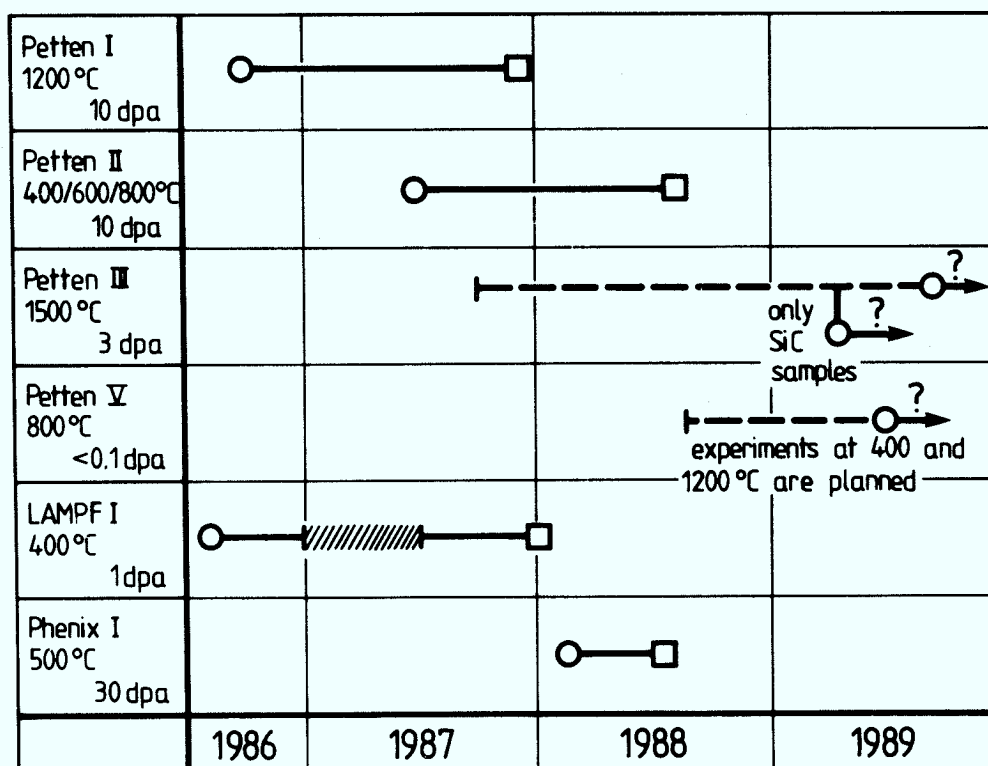
		Density g cm ⁻³	Bending strength N mm ⁻²	coeff. therm. expans. C · 10 ⁻⁶	grain size 10 ⁻⁴ m	Young's modulus N mm ⁻² × 10 ³	thermal conductivity W m ⁻¹ K ⁻¹
EK 98	Ri	1.85	50	3.1	12	12	70
FP 219	Sch	1.73	36	4.0		9	75
5890 PT	CL	1.83	55	4.7	20	11	108
S 1611	CL	1.76	64	5.5			70
S 1260	CL	1.68	52	6.3		8	123
V 1325	Ri	1.90	82	3.7	6	15	70
ASR-1RS	Si						
BN	ESK	2.2	45	4.4		23	30
Si ₃ N ₄	ESK	3.2	725	3.2		310	35
SiC + 5% AlN	ESK	3.22	450	4.6		310	80
SiC + 2% AlN	Hi		550	4.0		480	100
SiC + 2% AlN	ESK	3.2		4.1		90	
SiC + 2% BeO	Hi	3.2	470	3.7		460	270
Aerolor 05	CL	1.8	115	1.5 8.5 ⊥			225
Aerolor 46	CL	1.7	105				300 110 ⊥
FMI 4D 333-6	FMI						
CF 222	Sch	1.65	240			80	
SiC-SiC Oriented Pyrographite	Ga Fi						

Tab. 3: Nominal properties of materials irradiated in the present irradiation experiments of IRW; values taken from official documents of the producers or from publications; values are not averaged if they differ in different documents/publications but are taken arbitrarily; producers: see Tab. 1.

It shall be pointed out once more that the GRIPS test (Petten V) is primarily devoted to the thermal conductivity and its changes by neutron irradiations.

6. SCHEDULE AND STATUS OF THE IRRADIATION EXPERIMENTS

The first fusion relevant irradiation experiment of IRW was the LAMPF I experiment that started in 1986. The last one of the present series is planned to begin irradiation in 1989/early 1990. The real, resp. the nominal, time schedule for the six irradiation tests is given in fig. 19.



- : start-up of irradiation
- : end of irradiation
- → : planned start-up of irradiation
- ┤ — : originally planned start up
- //// : interruption of irradiation

Fig. 19: Time schedule and status of the first group of six fusion relevant neutron irradiation tests of IRW (partially together with or under guidance of partner institutions)

Four of the six irradiation experiments are already finished, at least as far as the neutron irradiation is concerned. Details of the post irradiation examination will be given in separate PIE-reports. It shall finally be pointed out that one important procedure for PIE will be the sample testing under high heat fluxes in an electron beam device in the Hot Cells of IRW which is presently under development, and in the electron beam device at SNLA (Sandia National Laboratories Albuquerque, USA), to investigate possible neutron effects on the thermal fatigue, thermal shock, and erosion behaviour of the materials.

7. CONCLUSIONS

A broad data base exists for the irradiation behaviour of carbon based materials, primarily nuclear grade graphites, that has its origin in the development of the High Temperature Gas Cooled Reactor. This experience allows an at least qualitative prediction of the irradiation behaviour of new graphite qualities that are developed particularly for fusion application. Moreover, the available information on the neutron induced changes of the relevant material properties and material damages localizes somewhat precisely those ranges of neutron fluence and irradiation temperature in which the irradiation effects are most critical.

These facts allow the conclusion that a limited number of well-defined irradiation experiments in fission reactors will provide a sufficient data base also for the new graphite qualities and other carbon based materials for fusion application. For heat-conducting ceramics like alloyed Silicon Carbides discussed in this paper some more systematic irradiation tests might be necessary.

The new irradiation test series of IRW described before is based on this conclusions and is therefore part of the neutron irradiation activities in the fusion community. As soon as new candidate materials are defined - e.g. by high heat flux tests in

electron beam or ion beam experiments, e.g. new CC qualities - they will be irradiated under the discussed conditions, too.

Although all irradiation data are based on irradiation tests in fission neutron spectra, the informations are directly valid for fusion conditions if the discussed "transfer factor" from fission neutron to fusion neutron damage effects proves to be sufficiently accurate or if an accurate "transfer factor" for the resp. material can be derived from the available data and if necessary by some particular experiments. In this case, there would be no urgent necessity for irradiation tests of the non-metallic materials in a new fusion-relevant neutron source which is presently under discussion.

This conclusion may be no longer valid when the role of transmutation products is examined. The maximum energy of 2 MeV for fission neutrons is not sufficient to generate transmutation products in low-Z non-metallics as has been discussed in this report. In this case some preceeding basic experiments would be necessary - and sufficient - to answer the question whether transmutation products play an important role for the lifetime of graphites, pyrocarbons, CC's, and heat conducting ceramics in the first wall of fusion devices.

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