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VISCOSITY MEASUREMENT OF FUEL SUSPENSION UNDER IRRADIATION BY MEANS OF VISIR-2 MEASURING DEVICE

by

A.M. SCHNEIDERS and G. HOLLEBECK

1972

Joint Nuclear Research Centre
Ispra Establishment - Italy
Materials Division

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ABSTRACT

Development and construction of VISIR-2 based on the experience with VISIR-1 are described. VISIR-1 and VISIR-2 are viscometers without rotating parts.

Through the use of second viscometer the viscosity of a liquid was been measured for the first time in a nuclear reactor. The liquid under investigation was a fuel suspension in water and was tested using the Ispra-1 reactor. The fuel was subjected to a thermal neutron fluence of about 0.9×10^{20} neutrons/cm^2.

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KEYWORDS

PLANNING
FABRICATION
VISCOSIMETERS
IN CORE INSTRUMENTS
MEASURED VALUES
FUEL SUSPENSIONS

CONTENT

ILLUSTRATIONS

VISCOSITY MEASUREMENT OF FUEL SUSPENSION UNDER IRRADIATION BY MEANS OF VISIR-2 MEASURING DEVICE. *)

1. INTRODUCTION

One of the problems encountered by the Dutch company, KEMA, in the development of a suspension reactor, is the stability of its fuel. The process which is believed to take place is that the fuel particles partly desintegrate and afterwards, due to chemical bondings, agglomerate again. This process influences the viscosity of the fuel. In order to study this effect, a contract was stipulated between KEMA and EURATOM which included the use of the facilities of the Ispra-1 reactor. A powder consisting of 98.5% ThO_2 particles and 1.5% UO_2 particles in water at a concentration of 200 grams per liter was employed as fuel suspension. The average particle-diameter is 5 μm and the Uranium in the fuel is 90% enriched.

A similar experiment has been carried out using VISIR-1 device whose working temperature was set at 200°C. The measuring device has been described in an earlier publication [1]. It is based on a mechanically vibrating system submerged in the fuel suspension. The attenuation of the system is a measure of the viscosity of the fuel suspension. The attenuation is obtained through the logarithm decrement.

The mathematical description of this technique will be published later [2]. For the present experiment the

*) Manuscript received on March 14, 1972

VISIR-1 apparatus had to be modified and the modified version will be known from here on as VISIR-2. VISIR-2 is shown schematically in figure 1.

2. SOME OF THE ESSENTIAL MODIFICATIONS OF VISIR-1

a) In order to keep the temperature constant during the irradiation process and during the reactor shut-down periods, a heating coil (Fig. 1-44) was placed around the fuel capsule. The working temperature was fixed at 300°C. This represents an increase in the working temperature with respect to VISIR-1 and it resulted in a higher pressure inside the capsule. In order to accomodate the higher pressure the capsule's wall-thickness had to be increased to 4 mm. However this resulted in a heavier capsule which required external springs with higher spring constants in order to keep the resonant frequency unchanged. Due to the fact that at maximum deflection the ratio of the stored energy of the internal springs to the stored energy of the external springs had to be kept at 0.6, it was also necessary to increase the weight of the float.

b) A rupture disc (Fig. 1-17) which is present for security reasons was this time welded instead of pinched, as previously done. This disc was welded by means of the electron beam method, but this method initially caused some cracks in the welding area (Fig. 2). The cracks were caused by the rapid cooling of the material after the welding process and also because the capsule and the rupture disc were made of diffe-

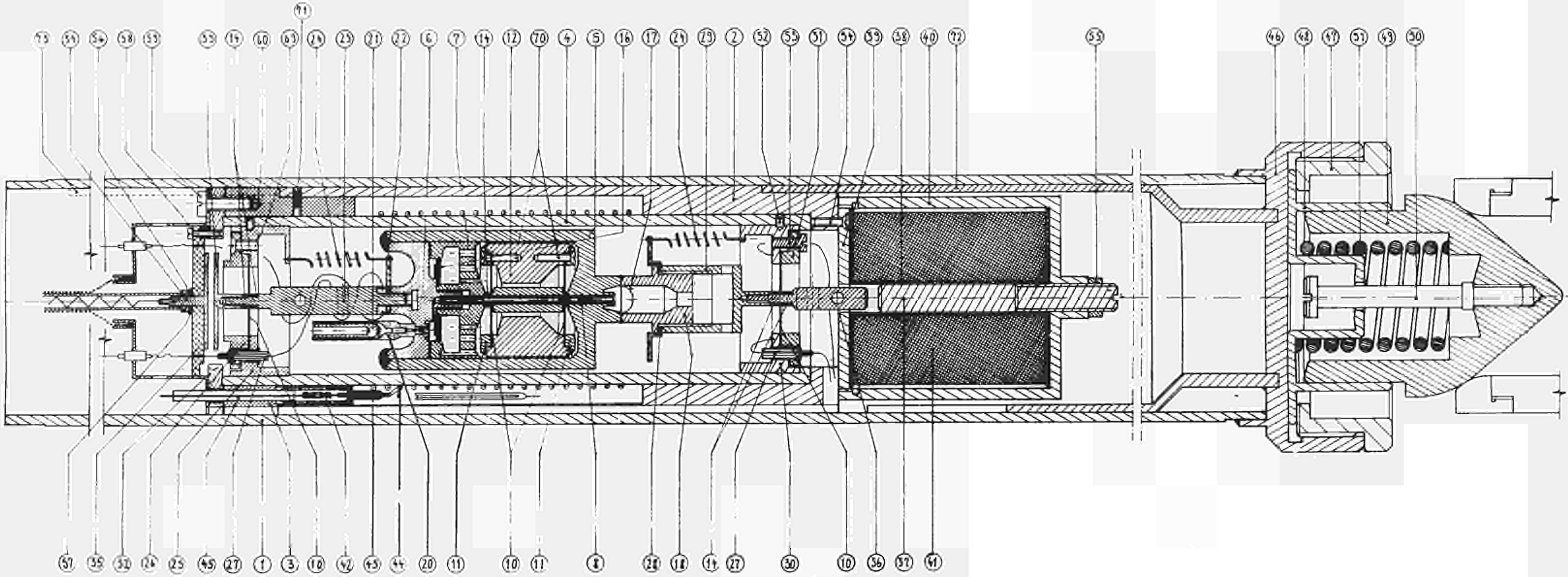

Fig. 1 Schematic diagram of VISIR - 2

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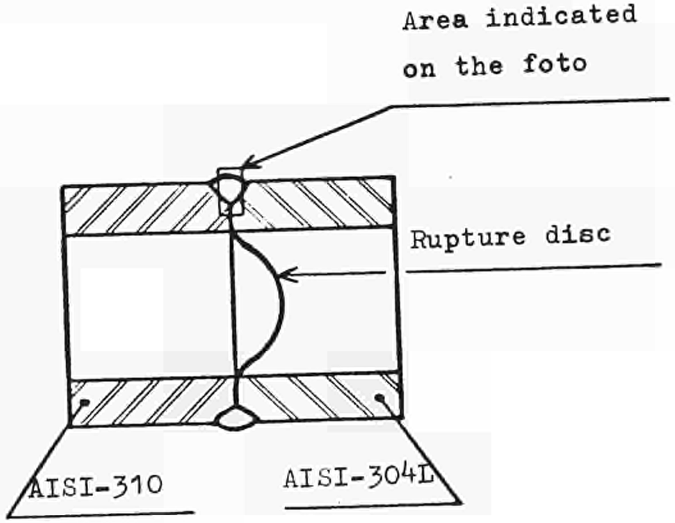
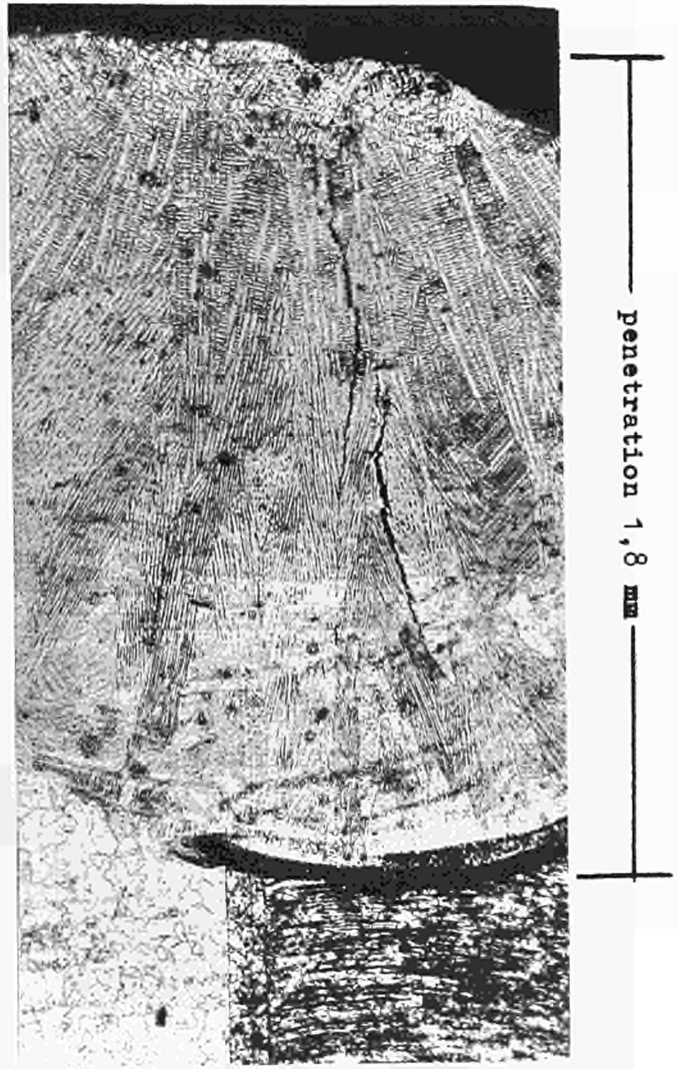
VISIR-2

Welding of rupture disc
by electron beam process





Enlargement of the foto 60x

Figure 2. Cracks in the joint of the rupture disc of the capsule

rent stainless steels. In fact the capsule was made of A.I.S.I. 310 since it has excellent mechanical properties at high temperature, while the disc was made of A.I.S.I. 304L since A.I.S.I. 310 is not produced in thin sheets.

The problem of cracks-formation appeared because of the different behaviour of the steels employed. A.I.S.I. 310 requires a stress relief treatment at high temperature after welding; on the other hand A.I.S.I. 304L must be cooled rapidly in a certain temperature range in order to avoid intercrystalline precipitation which lends itself to stress corrosion. A proper solution for the problem of crack-formation was achieved by

i) fabricating the piece which comes directly below the disc, namely the support for the lower helical springs (Fig. 1-18) (Fig. 3), of A.I.S.I. 304L and

ii) by applying a thermal treatment directly after welding for 10 minutes while the temperature is decreasing. Kema has shown that a special type of argon-arc welding could have also been used to solve the problem of crack-formation [3].

c) The migration of the fuel particles to the rupture disc through the central tube (Fig. 1-8) has been prevented by placing a filter (Fig. 1-16) in the upper part of the expansion area. The necessity of this filter was suggested by the experience obtained in the agitated high temperature experiment performed at Mol, Belgium, using the high-flux reactor BR2. It may be recalled that a large amount of the fuel particles had vanished during the experiment [4]

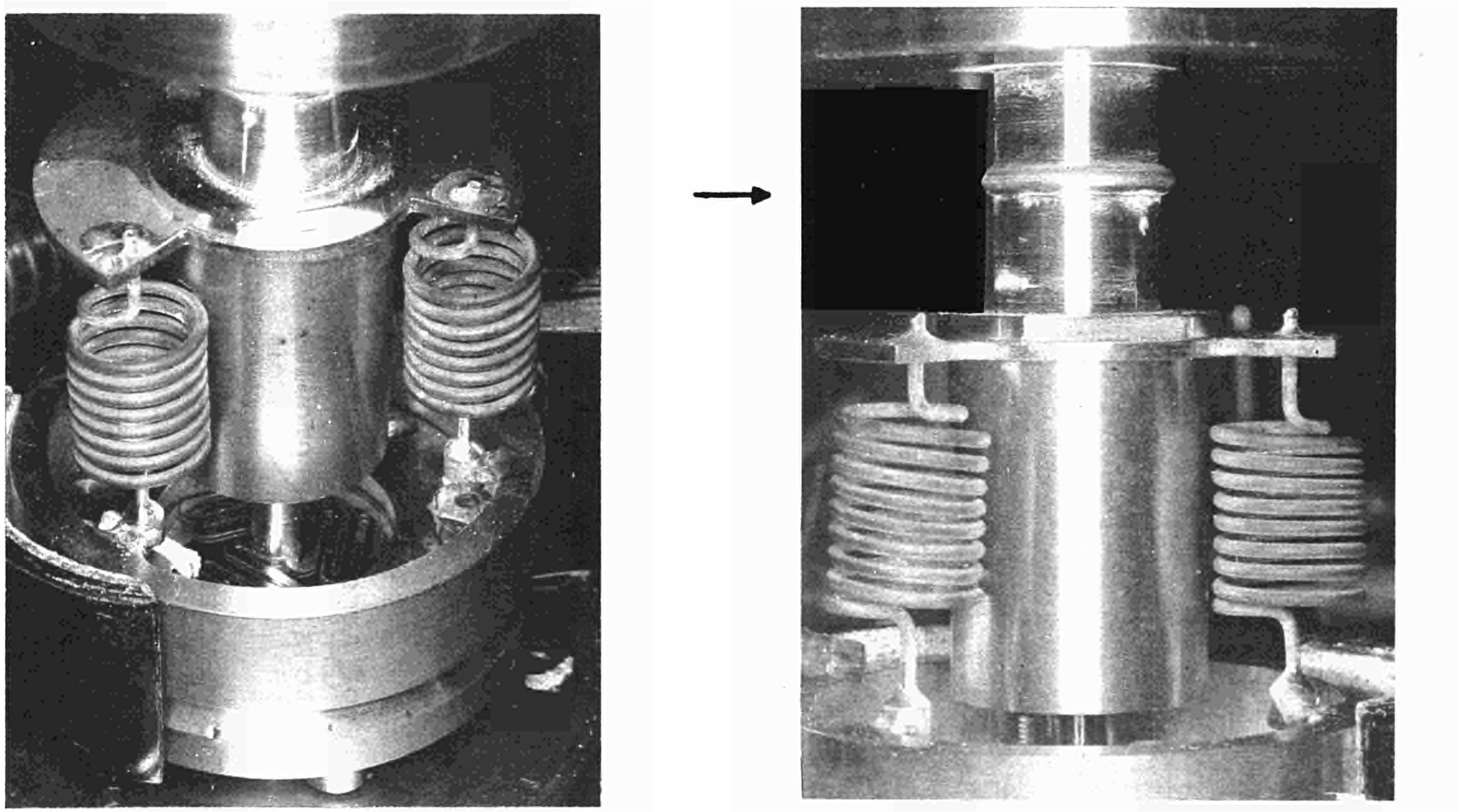


Figure 3. Lower helical and flat springs
Arrow indicates the rupture
disc joint.

d) Contrary to what had been agreed earlier it was decided not to place in the capsule a Al-Co monitor wire for the measurement of the integrated thermal neutron flux since the water may corrode the aluminium in the wire when the capsule is heated.
The omission of the monitor wire did not cause a loss of information since the received flux can be calculated by means of the Co-content in the float springs. A chemical analysis of the composition of the spring-material gave the following result:
C: 0.035%; Cr: 19.55%; Ni: 59.40%; Co: 16.55%.

3. THE MEASURING SYSTEM

The fuel capsule is mechanically excited by an electromagnetic system which consists of a coil (Fig. 1-41) and its inner core (Fig. 1-36). During the excitation the capsule oscillates sinusoidally with respect to the fixed rig and with respect to the vibrating float in it. When the current through the coil is kept constant the amplitude of oscillation of the capsule increases as the driving frequency gets closer to the resonant frequency. If at resonance the excitation is interrupted, the amplitude gradually decreases with time while the oscillatory motion is still sinusodial. The damping is defined as the logarithm of the ratio of the amplitudes of two successive oscillations. In reality, however, for this work the damping is found by measuring the mean value of the logarithmic decrement, during the interval of time in which the amplitude decreases from its maximum value to about 2% of this value. This measuring procedure allows the observation of how the viscosity varies

with the shear velocity of the liquid since the damping is a mesure of the viscosity while the amplitude is a measure of the shear velocity.
An additional consequence of the above mentioned method is given by the fact that the time of a measurement varies inversely with the damping. The damping depends on the viscosity and on the geometry of the system. The geometry of the system must chosen so that the measuring time is shorter than the time needed for the fuel particles to precipitate. This is an important feature which must be carefully taken into account when the measuring system is built. The longest measuring time which can be used for the VISIR-2 viscometer, without incurring precipitation, corresponds to a viscosity value of 0.09 centipoise, while the largest value for a measurable viscosity is 2.0 centipoise. Figure 4 is a graph which has been employed to translate the damping to its corresponding viscosity value.

4. PERTINENT TECHNICAL DATA

Flat springs (Fig. 1-10)

material: Nimonic 90; composition: 0.035% C, 19.55% Cr, 59.40% Ni, 16.55% Co

Helical springs (Fig. 1-24)

material: Nimonic 90; wire diameter: 1.2 mm; winding diameter: 11.4 mm; number of windings : 8.3 number of springs: 6

Capsule (Fig. 1-5)

material above the rupture disc: A.I.S.I. 310; material below the rupture disc:

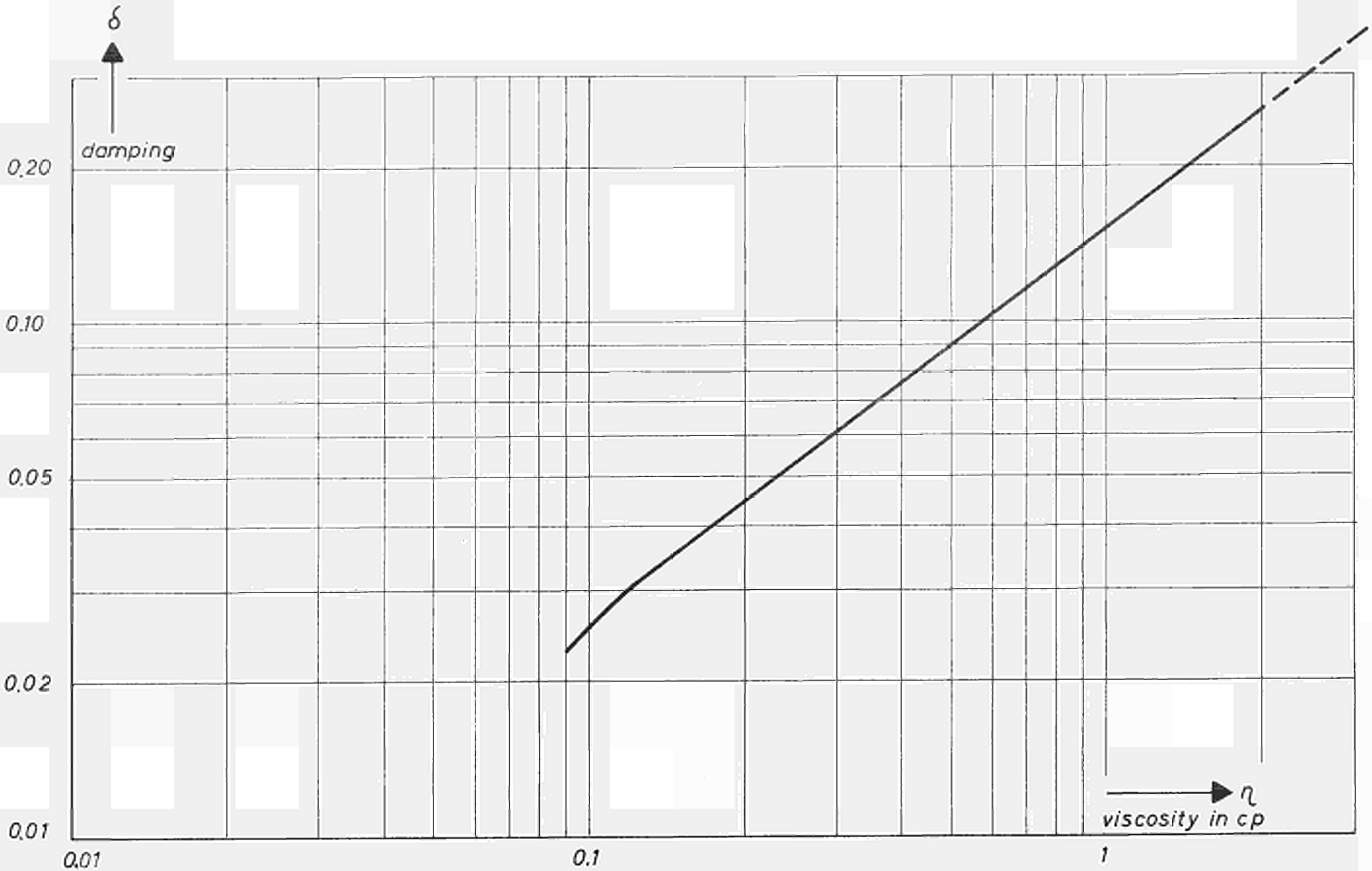


FIG. 4 DAMPING VERSUS VISCOSITY

A.I.S.I. 304L; wall thickness: 4 mm;
total weight: 955 grams

Float (Fig. 1-12)

material: A.I.S.I. 310; weight: 256 grams

Filter (Fig. 1-16)

material: sintered stainless steel;
average pore diameter: 3 μm

Amplitude of float movement

$\leqslant$ 1.2 mm peak to peak

Amplitude of the capsule movement

$\leqslant$ 2.0 mm peak to peak

Horizontal amplitude of the float movement

$\leqslant$ 0.2 mm peak to peak

Viscosity range

0.09 centipoise to 2.0 centipoise

Working temperature

300°C

Maximum admittable temperature

310°C

Measuring frequency

17 Hz

Total number of oscillations during the experiment

$\geqslant 10^8$

Free capsule volume after mounting

24 cm^3

Capsule filling

13.5 grams demineralized water; 2.700 grams mixed oxide powder (98.5% ThO_2, 1.5% UO_2); 0.336 grams paladium; a platinum wire with a diameter of 0.5 mm and a length of 10 cm; the wire is covered with Pt-black; hydrogen pressure: 4 atmospheres at room temperature.

Enrichment of the uranium

90%

Gap between the magnet and its core

4 mm

Thermocouples positions

thermocouple n. 1: the condensor plate
thermocouple n. 2: the capsule
thermocouple n. 3: the fornace
thermocouple n. 4: the lower flat springs
material: Chromel-Alumel

Distance between condensor plates (Fig.1-33)

2.1 mm

Coil (Fig. 1-41)

1340 windings of anodized aluminium wire; wire diameter: 1 mm

Helium pressure in the rig

1.04 atmosphere

Rupture disc (Fig. 1-17)

material: A.I.S.I. 304L; thickness 0.19 mm; mean rupture pressure at 20°C: 560 atmospheres; mean rupture pressure at 300°C: 375 atmospheres

Welding procedure for the rupture disc

welding time: 4 seconds; subsequently heat treated: 40% beam intensity during 90 seconds, 30% beam intensity during 210 seconds, 20% beam intensity during 180 seconds.

Heating coil (Fig. 1-44)

Thermocoax Philips; maximum power: 2kW

Irradiation time

65 days

5. CHRONOLOGICAL DEVELOPMENT AND EXPERIMENTAL PROCEDURE

September 11, 1969

Signature of the contract of VISIR-2.

October 23, 1969

Opening of the budget in Ispra for VISIR-2 and official start of the activities.

October 1969 - April 1970

Introduction of Nimonic-90 as spring material. Development of a technique to fabricate the flat springs. Nimonic is cut in strips of about 30x5 cm^2. These strips are alternatively annealed at 1080°C and cold rolled untill the desired thickness is achieved. In the last cold-rolling process a 30% deformation is obtained. In order to flatten the strips a tensile machine is used while the hardening is obtained by heat-treating the samples for 4 hours at 650°C. In order to avoid buckling the specimens are held under compression during the heat treatment. A laboratory model of the viscometer is designed, is fabricated and is calibrated. The rig is built in the central workshop of the center and simultaneously, creep and tensile properties of the springs are determined.

May 1970

At a conference between some representatives from the Kema company and Euratom it is decided that before the in-pile experiment, a dummy rig will be made to carry out a simulated test. The dummy rig will be filled with natural mixed oxide powder and will have a following goals:

a) to observe whether or not the dummy rig would last for at least six weeks.

b) To determine the mechanical wear of the fuel particles on the inner surface of the capsule and on the springs.

c) To observe the fatigue properties of the springs at the working temperature.

d) To study the behaviour of the fuel particles inside the capsule.

June 5, 1970

The rupture disc of the dummy rig breaks unexpectedly. It is concluded that the disc must be made of thicker sheets to sustain a pressure up to 375 atmospheres at 300°C and plans are made for the building of a second dummy rig.

July 8, 1970 - September 10, 1970

Measurements on the second dummy rig succeed. At the end of the test, the capsule is sent to Arnhem, Holland, in order to study possible wear damage. In a subsequent meeting it was decided to build, as soon as possible, an actual rig making use of the information gathered during the simulated tests.

September 1970 - October 1970

The welded joint of the rupture disc cracks unexpectedly which causes the water to drain out of the capsule. This mishap brings about an extensive research to determine the causes of these cracks and the best solution to avoid them.

November 1970

It is discovered that earlier welded joints had similar cracks. Experts from the Kema and Euratom conclude that the cracks are due to material contraction during the rapid cooling after welding.

December 1970 - March 1971

In order to determine why cracks are created at the joints several different welding procedures are studied. Figure 2 shows that craks sometimes terminate just below the surface and this makes them hard to detect. A welding procedure has been developed which can be used to avoid crack-formation. This welding procedure is nothing else than a normal electron beam method. However care has been taken in choosing the proper welding velocity, welding depth and the right heat treatment after the welding process, combined with the right choice for the material for the lower part of the capsule.

March 9, 1971

The decision to build the second VISIR-2 reactor rig, applying the new welding procedure for the rupture disc, is reached.

March 10, 1971 - May 12, 1971

The second VISIR-2 reactor rig is built and its calibration curve is determined (Fig. 4).

May 22, 1971

VISIR-2 is placed into position 6 in the ISPRA-1 reactor.

May 25, 1971

The instrumentation of the VISIR-2 viscometer is tested.

May 26, 1971 - 9.00 p.m.

ISPRA-1 reactor is put into operation.

May 26, 1971 - 11.00 p.m.

The reactor power reaches 3 MW. The capsule temperature climbs to 300°C and is still rising.

May 26, 1971 - 11.18 p.m.

The reactor power is diminished to 2.5 MW in order to prevent overheating of the VISIR-2 capsule.

May 27, 1971

Since the reactor power at normal operative conditions is 5 MW and since the working temperature of the capsule is reached when reactor power is only 2.5 MW, different methods are discussed to limit the capsule temperature at 300°C and still allow the power of the reactor to reach its operative level. Oxidizing the outer walls of the capsule in order to raise the heat exchange brings no improvement. Since position 4 happened to be farther from the fuel elements, its is decided to place VISIR-2 into position 4.

May 28, 1971 - 3.05 a.m.

Reactor is shut-down.

May 28, 1971 - 5.35 a.m.

VISIR-2 is moved into position 4 and the reactor is put again in operation.

May 28, 1971 - 8.55 a.m.

Dr. Hasenjäger, Head of the reactor-section, propose to remove VISIR-2 because the reactor power cannot be raised above 2.6 MW without overheating the VISIR-2 capsule.

May 28, 1971 - 9.35 a.m.

Dr. Marchetti, Head of Materials Division, suggested to Dr. Hasenjäger that even though the reactor power was only half of its normal output, the VISIR-2 experiment should be tried anyway. After subsequent consultation of Dr. Marchetti with Dr. Hasenjäger together with officials involved with other experiments in the reactor, it was decided to do so.

May 28, 1971 - June 4, 1971

Regular measurements with VISIR-2 are made.

June 4, 1971 - 8.07 a.m.

The reactor is shut-down and VISIR-2 is finally removed.

June 1971 - July 1971

Preparations are made to introduce VISIR-2 in position 6 EV, where the capsule can be placed about 60 cm below the center of the core of the reactor. At this level the gamma heating and the neutron flux are considerable lower than those in the core and it is hoped that by placing the capsule there the reactor can operate at normal power, and that the overheating of the capsule can be avoided.

August 17, 1971

VISIR-2 is put in position 6 EV 55 cm below the core.

August 23, 1971 - 8.50 a.m.

The reactor begins to operate.

August 23, 1971 - 2.35 p.m.

The power of the reactor is 4,2 MW while the capsule temperature is already 298°C. It is decided to lower the capsule another 6 cm.

August 24, 1971

The reactor is shut-down. The capsule is put 61 cm below the core and reactor is again started.

August 24, 1971 - 9.40 p.m.

Reactor power reaches 4,4 MW and with this amount of power the capsule is 293°C.

August 25, 1971

The reactor has been shut-down for again 2 more hours for security reasons related to another experiment. When the reactor is started again the capsule temperature is 304°C while the reactor's power is only 4.15 MW.

August 26, 1971

It is believed that the helium in the rig is contaminated with air and that this causes a lowering of the heat exchange rate between the capsule and rig wall. It is decided to clean the helium by the spooling process. After this additional procedure the reactor power can be raised to 4.7 MW while the temperature of the capsule decreases to 296°C.

August 26, 1971 - September 19, 1971

Regular running of rig VISIR-2. On 30 August it is discovered that the furnace is short-circuited and that the monitoring thermocouple is burned out. Since the working temperature of the capsule was achieved without using the furnace its malfunctioning had no bearing on the experiment. On September 13 there is a reactor shut-down for reasons which are not related to the running of VISIR-2 experiment.

September 19, 1971 - September 22, 1971

The reactor is shut-down for normal operative procedures.

September 22, 1971

The reactor is again started and its power is allowed to reach 5 MW.

September 22, 1971 - October 31, 1971

VISIR-2 rig is functioning normally and the experimental data are taken at the proper intervals of time.

October 31, 1971

Periodical shut-down of the reactor.

November 7, 1971

Removal of VISIR-2 from the reactor as requested by Kema.

6. NEUTRON FLUENCE

Figure 5 gives the unperturbed neutron flux distribution along the vertical axis for position 10 of the ISPRA-1 reactor. The integrated flux can be evaluated by assuming that the same distribution is valid also for position 4,6 and 6 EV and that the flux is proportional to the reactor power. If a factor 0.7 is taken as the attenuation factor, due to the steel walls, a fluence of about 0.9×10^{20} neutrons/cm^2 is obtained during the entire irradiation period. The above value will be verified by activation measurement of the cobalt content present in the internal springs.

7. DISCUSSION OF RESULTS

Figure 6 represents the measured viscosity as a function of irradiation time. The irradiation was not continuous because it was interupted several times. The longest interruption took place after 220 hours of operation when the rig was removed from the reactor in order to be modified and then placed in position 6EV. This interruption lasted for eighty days. A second interruption accured after 880 hours of irradiation and it lasted three days. All other reactor shut-down periods, which were much shorter, are considered to be part of the irradiation time.

Figure 6 shows that the viscosity increases with irradiation time. One can observe that the viscosity remains fairly constant during the first 300 hours of irradiation while a marked increase took place between 300 and 800 hours. After 800 hours the viscosity value did not change

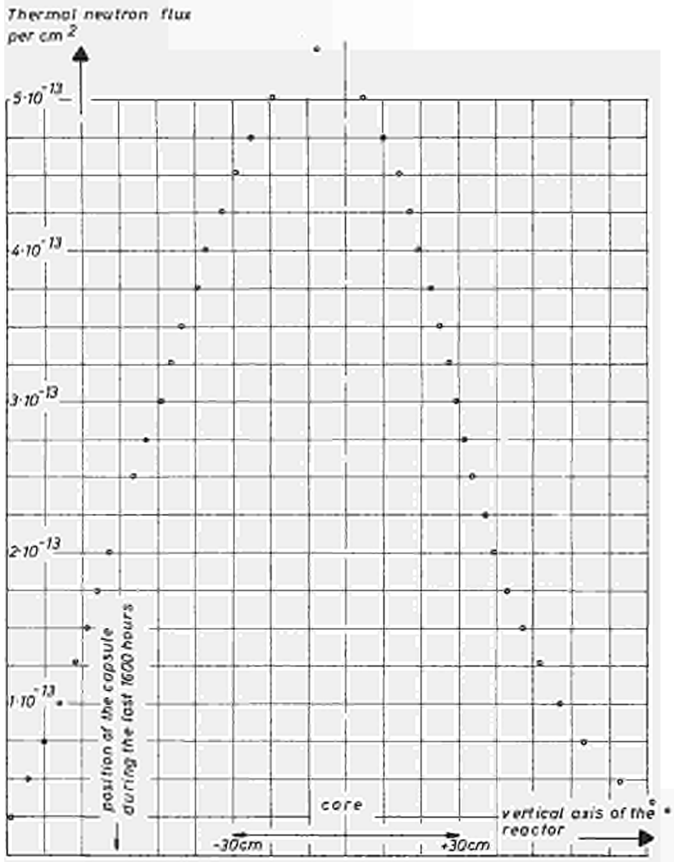


FIG. 5 THE IMPERTURBED FLUX ALONG THE VERTICAL AXIS

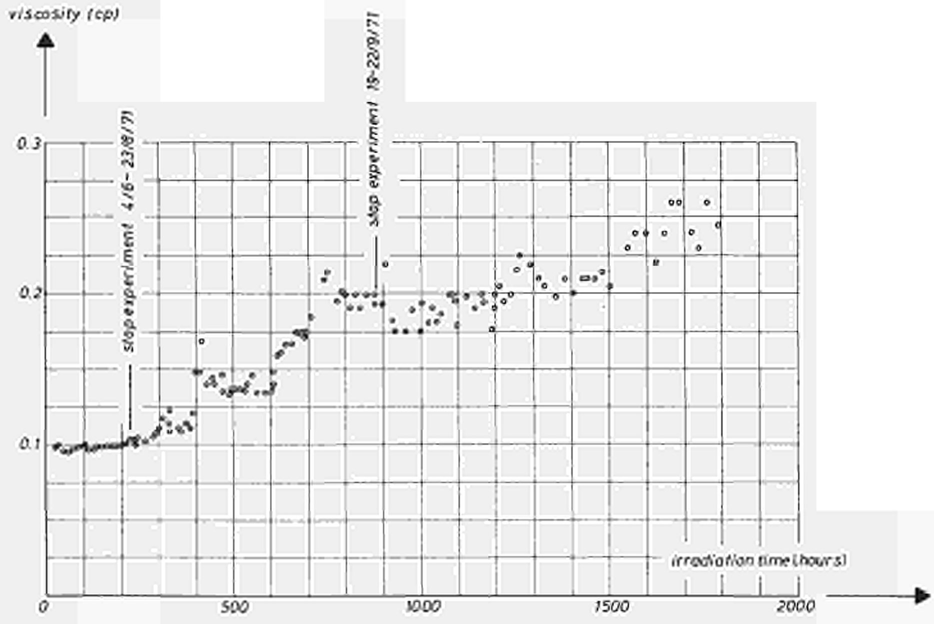


FIG. 6 VISCOSITY VERSUS IRRADIATION TIME

so markedly. It is noticed that as soon as the viscosity increases the dispersion of the data increases also. The dispersion can be traced to the fact that with increasing irradiation time the behaviour of the fuel suspension differs increasingly from that of a pure newtonian liquid. This implies that the viscosity becomes dependent upon the vibrational amplitude of the capsule. Consequently the viscosity varied slighty during a single measurement. The computed values showed a lower reproducebility.

A second factor of minor importance which did influence the incertainty of the measured values of viscosity was due to the following reason: the capsule had to be lowered nine days after the beginning of the experiment because, as described earlier, the capsule would become overheated as the reactor power was allowed to reach its normal level. This lowering of the capsule resulted that a section of the measuring cables together with the thermocouple wires were exposed to the maximum radiation flux. The capsule was kept in the above mentioned position for the next 65 days of the experiment. After a while it was noticed that the insulation of the measuring cable which was made of silicon oxide fibers has deteriorated due to possible radiation damage. The decreasing insulation did not effect the viscosity measurements but it did diminish the amplitude of the response signal thus increasing slightly the uncertainty of the data.

In figure 7 the readings of two thermocouples as they appear on the control desk against the same time scale as that of figure 6 are plotted. Thermocouple number 1, was mounted on the fixed condensor plate (Fig. 1-35). It was of the thermocoax type and its insulating sleeve was made of aluminia powder. This thermocouple was

unaffected by irradiation. In figure 7 the sudden rise in the temperature, after 220 hours, is due to the change of position which implies a different distribution of the gamma heating on the viscometer.
The thermocouple number 2 measured the temperature of the capsule and consequently vibrated with it.
For this reason a thermo-coax thermocouple could not be used on the capsule. Therefore the chosen thermocouple had the same insulation as that of the measuring cable and as it has been discussed earlier this insulation, unfortunately, suffered irradiation damage. This caused the thermocouple to indicate an apparent decrease in temperature during the second half of the experiment. Here, it must be mentioned that thermocouples 3 and 4 failed during the experiment.

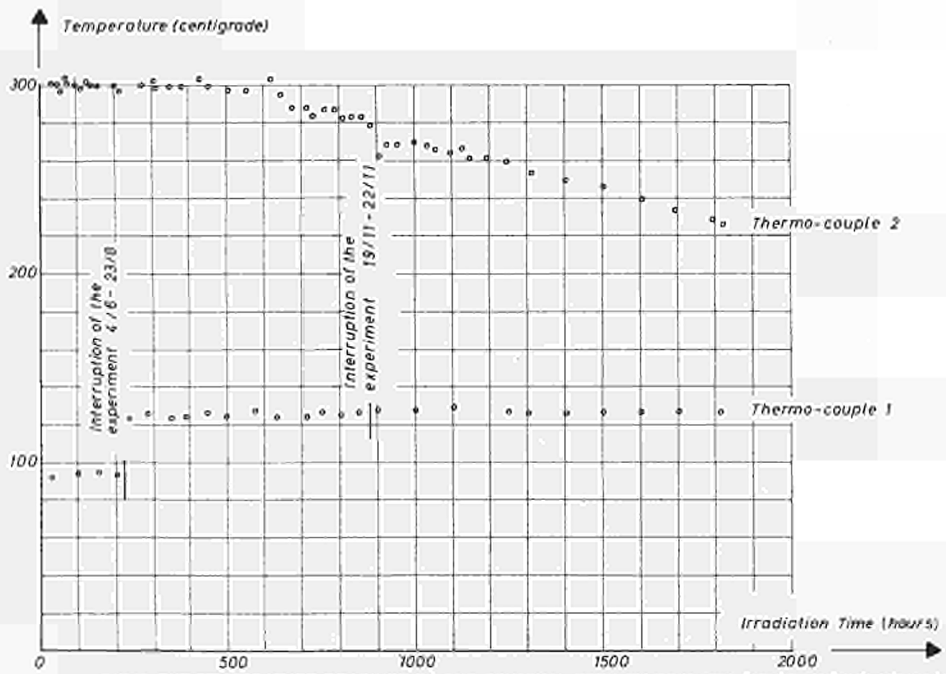


FIG. 7 THE TEMPERATURE READINGS ON THE CONTROLL DESK BY TWO DIFFERENT THERMO-COUPLES

8. CONCLUSION

The VISIR-2 experiment has shown that it is possible to construct a viscometer without rotating parts and to do viscosity measurements with it, in a nuclear reactor. More precisely it is found that the proper functioning of the developped viscometer is not affected by radiation damage when it is exposed to radiation flux for a period of time that can be as long as a few months. The eight flat springs (Fig. 1-10) which we believed to be the weakest point of the viscometer and the six helical springs (Fig. 1-24) sustained vibrations of the order of 10^8 cycles without break-down up to the end of the experiment. This demonstrates that the mechanical properties of the springs were not adversily affected by possible radiation damage. Neither was the youngs modulus essentially influenced by radiation. In fact, the resonant frequency varied only about 1.5% from its original value and this variation is only partly due to the change in youngs modulus. The calibration curve was left unchanged by this small increase in frequency. The difficulties encountered with the measuring cable and the thermocouples were not so serious to influence the experimental results and they can be avoided in a similar experiment in the future.

During the experiment the measured viscosity of the fuel suspension increased as a function of the neutron fluence. At the beginning of the experiment the viscosity corresponded to that of the pure water (0.1 centipoise at 300°C), while at the end the value had more than doubled to about 0.25 centipoise. It is thought that this increase in the viscosity is due to a change of the fuel particles under irradiation.

To what percentage the desintegration of the fuel particles are responsible to an increase of 0.15 centipoise in the viscosity is a question still left open. It is hoped that the post - irradiation analysis shall provide an answer to this question.

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