Metallurgy Department. Progress report for the period 1 January to 31 December 1976

Research Establishment Risø, Roskilde

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Metallurgy Department Progress Report

for the period 1 January to 31 December 1976

March 1977

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Metallurgy Department
Progress Report

for the period 1 January to 31 December 1976

ABSTRACT

The activities of the Metallurgy Department at Risø during 1976 are described. The work is presented in four chapters: General Materials Research, Technology and Materials Development, Fuel Elements, and Non-Destructive Testing. Furthermore, a survey is given of the department's participation in international collaboration and of its activities within education and training. A list (with abstracts) of publications and lectures by the staff during 1976 is included.

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INTRODUCTION

A political decision about the establishment of nuclear power stations in Denmark was expected in 1976 and a number of preparatory studies were carried out. Around midsummer the Government, however, decided to postpone the decision, mainly because of uncertainty about the deposition of the nuclear waste. This postponement has not influenced the programmes at Risø to any large extent since the establishment still must be in a stand-by position with respect to the nuclear field.

A major change in the status of the Research Establishment Risø took place in 1976: the Atomic Energy Commission, hitherto responsible for Risø, was abolished, and a Risø Board was set up as the responsible body for the establishment. The official name of the establishment was changed accordingly to Research Establishment Risø (Danish Atomic Energy Commission being omitted). Also, Risø was transferred from the Ministry of Education to the Ministry of Commerce and Industry.

In the internal Risø organization the hot cell facilities were transferred from the DR 3 Reactor Department to the Metallurgy Department.

The technical programmes of the department were continued within such areas of nuclear technology as design and fabrication of fuel elements, in- and out-pile testing, post-irradiation examinations, and non-destructive testing of nuclear components. To supplement these programmes, work was done in the field of general materials research, including studies of mechanical properties, structure, radiation damage, and corrosion. After an assessment of the possible application of its knowledge to research in the field of alternative energy technology, the department has defined the following three areas as being relevant:

Fig. 1. A view of the Risø stand at the Intertool exhibition.

a) high temperature materials
b) fibre reinforced composites (e.g. for windmill rotors) and
c) metal-hydrogen systems (e.g. for transport and storage of hydrogen).

The development of a tube inspection system for defect and dimensional control of tubes (e.g. tubes for fuel element cladding) was completed. The development was carried out in collaboration with the Elsinore Shipyard Ltd., which is now marketing the equipment together with Krautkrämer GmbH., Germany. Another project brought to the marketing stage, was the development of

a fuel pin performance code, Hotcake/TR, also in collaboration with the Elsinore Shipyard.

In the present report the work in the department is presented in four chapters: General Materials Research, Technology and Materials Development, Fuel Elements, and Non-Destructive Testing.

Work was also done under contract for industries and utility companies in Denmark and abroad. Due to their proprietary nature, most of these activities are excluded from the present report. Among the major areas of activity were fuel element development, high temperature components for the chemical industry and acoustic emission for non-destructive testing purposes (the latter in collaboration with the Danish Welding Institute). Other projects were centred on the development of materials and processes; in particular a number of problems related to brazing were solved. Post-irradiation examinations of irradiated materials and fuels were continued; extensive efforts were devoted to the examination of full-scale power reactor fuel rods ($Zr-UC_2$ and $Zr-UO_2-PuO_2$) and to isotope analysis.

Three senior staff members acted as part-time consultants to specific companies. In order to demonstrate its techniques and results to the public the Metallurgy Department participated in the Intertool Exhibition ir Copenhagen. In connection with the exhibition, lectures were given on selected topics within the range of activities of the department.

The department participated in international collaboration in a number of areas such as fuel element modelling, materials development and examination, and safety analysis. Furthermore, we are represented in a number of international projects and study groups under the auspices of the NEA, IAEA, EEC and of various Nordic organizations.

Educational activities were continued, students and post-graduate research workers from Denmark and abroad studied in the Department. The degree of tekn.dr. was conferred on one of the staff members.

One staff member received a prize from the Danish Welding Society for contributions to brazing research, and two others received, from the same society, an award for an article on welding of zircaloy.

GENERAL MATERIALS RESEARCH

Effect of Grain Size on Flow Stress of Aluminium

Tensile stress-strain data over a strain range from 0.2 to 30% were obtained at room temperature for 99.999% and 99.5% aluminium as a function of grain size. The yield-stress/grain-size relationship can be expressed as a Petch-Hall relation with approximately the same slope for the two materials. The flow-stress/grain-size relationship was analyzed in terms of matrix strengthening and grain boundary strengthening, and at intermediate strains this approach gives a good description of the effect of strain, grain size and purity on the flow stress.

Additive Strength Contributions

Copper with Al_2O_3 particles was used in this study of the effect of grain boundaries and particles on strength. A systematic investigation of the method of internal oxidation for fabrication of Cu-Al_2O_3 resulted in a reproducible procedure based on oxidation with a CO/CO_2 gas mixture. The size of the Al_2O_3 particles can to some extent be varied by this method.

Work Hardening in Fibre Composites

The work hardening of fibre composites was studied in the model system of copper with long tungsten fibres. From tension-compression cyclic tests, two important strength contributions were identified: the mean stress from the geometrically necessary dislocations at the fibres, and the contribution from the reduced spacing between the fibres caused by the accumulated dislocations. The mean stress at $-196^{\circ}C$ is in quantitative agreement with theoretical estimates, while the mean stress at room temperature is lower than estimated, and it is suggested that a reversible relaxation takes place by one or several hindered dislocation processes. The second strength contribution is not in agreement with a simple model for the process, but the numerical values are of the same order of magnitude as those derived from the model.

A study was performed of the strain parameters that are relevant to tensile experiments; they were related to the geometry

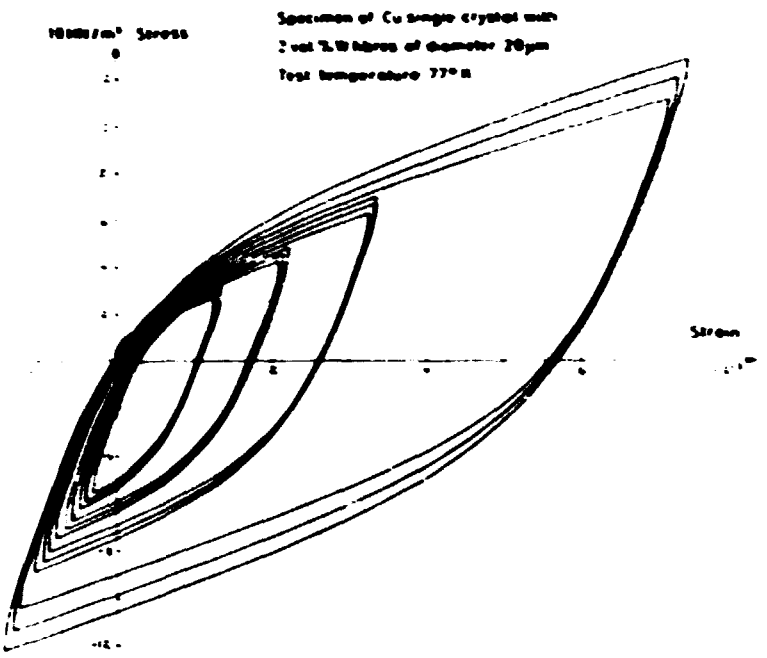

Fig. 2. Results of a cyclic test (Bauschinger test) performed on a fibre composite of Cu single crystal matrix reinforced with W fibres. From such tests in tension and compression the mean stress from the geometrically necessary dislocations can be determined independently of other stress contributions.

of the deformation and to the stress-strain curve of two-phase materials.

In a theoretical study of internal stresses in two-phase materials, the existing theory for the mean stress was extended to materials containing large volume fractions of inclusions with elastic constants different from those of the matrix. This led to a unified theory for the mean stress, the effective elastic constants and the effective coefficients of thermal expansion for fibre composites and dispersion-strengthened materials.

Creep in Fibre Composites

The model for the relation between the creep law of the matrix and the (resulting) creep law for the composite with short parallel fibres was extended to include the case of creeping fibres. The contribution from the matrix to the creep strength of the composite was also included in the model.

Rolling Texture of Copper

The rolling texture of unirradiated and irradiated copper was measured by neutron diffraction in order to examine the possible effect of irradiation on the transition from the copper-type to the brass-type texture.

Finite Element Model of the Deep-Drawing Process

A study was initiated to examine the deep-drawing process and other metal-forming processes of the same nature, and an axisymmetric finite element computer program was developed. The program takes into account the geometrical and material non-linearities arising from the large plastic deformations that take place during metal-forming processes.

Acoustic Emission

Studies were performed of the role of carbide failure as a source of acoustic emission in steels. Carbide failures were measured as a function of the applied tensile strain in a low-carbon steel containing pearlite colonies. The results indicate that carbide failures themselves are not resporsible for the recorded emission in this material. In a high-carbon steel, carbide cracking could be a source of acoustic emission. A model for the cracking of a carbide plate in a ductile matrix was developed, and the values of the energy released during cracking were estimated.

Acoustic emission was also applied to measurements on fuel-tubes under stress corrosion, and a general facility for recording of acoustic emission during tensile loading experiments was built.

Irradiation and Mechanical Properties

The effect of neutron irradiation on the stress-strain curve of single crystals was studied in pure copper, copper with aluminium in solid solution, and copper with dispersed Al_2O_3 particles. The stress-strain curves exhibit an extended region of yield at a fairly high stress level.

Irradiation creep experiments were made on preannealed nickel at 185°C. Electron microscopy of a specimen irradiated to a dose

of 1.5×10^{20} n/cm^2 under a stress of 130 MN/m^2 showed a structure with black dots in a density of approximately 10^{17} cm^{-3} and resolvable ($\gtrsim$ 70 Å) Frank loops in a density of approximately 4×10^{15} cm^{-3}.

Irradiation creep experiments at 185°C on zircaloy-2 showed that "black dots" are formed during the primary stage in densities of the order of 10^{16} cm^{-3}.

Radiation Damage in Stainless Steel

(In collaboration with Metallurgy Division, AERE Harwell, UK)

Studies of void nucleation in stainless steel of type 316 were continued with experiments in the high voltage electron microscope. Possible mechanisms for void shrinkage during irradiation were analyzed. Pipe-diffusion of vacancies from or of interstitials to the voids can explain the shrinkage behaviour observed during the experiments.

Radiation Damage in Pure Copper

(In collaboration with Metallurgy Division, AERE Harwell, UK)

Void nucleation in pure copper after irradiation at low temperatures (50-60°C) followed by annealing was studied in the electron microscope and by the positron annihilation technique.

Radiation Experiments on Copper-Nickel Alloys

(In collaboration with Metallurgy Division, AERE Harwell, UK)

The structure of the rectangular dislocation loops formed in copper-nickel alloys (1-20 wt% Ni) during electron irradiation in the high voltage electron microscope was studied by the weak beam technique, and the experimental images were compared with computer-simulated images. The loops were found to take up a Hirth-lock configuration, consisting of two Shockley partial dislocations and one stair-rod dislocation with Burgers vector a/3 <100>. The growth rate of dislocation loops in various copper-nickel alloys at different temperatures was measured with the aim of finding the binding energy between Ni atoms, or clusters of Ni atoms, and vacancies or interstitials.

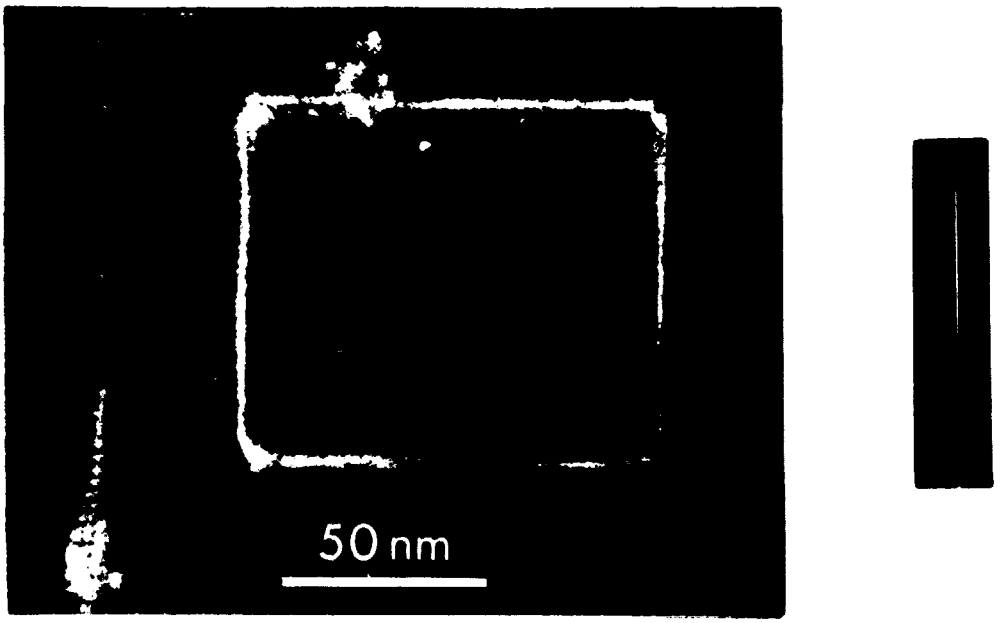

Fig. 3. Weak-beam electron micrograph of a rectangular loop in electron-irradiated Cu-10%Ni and computer-simulated image of the right edge.

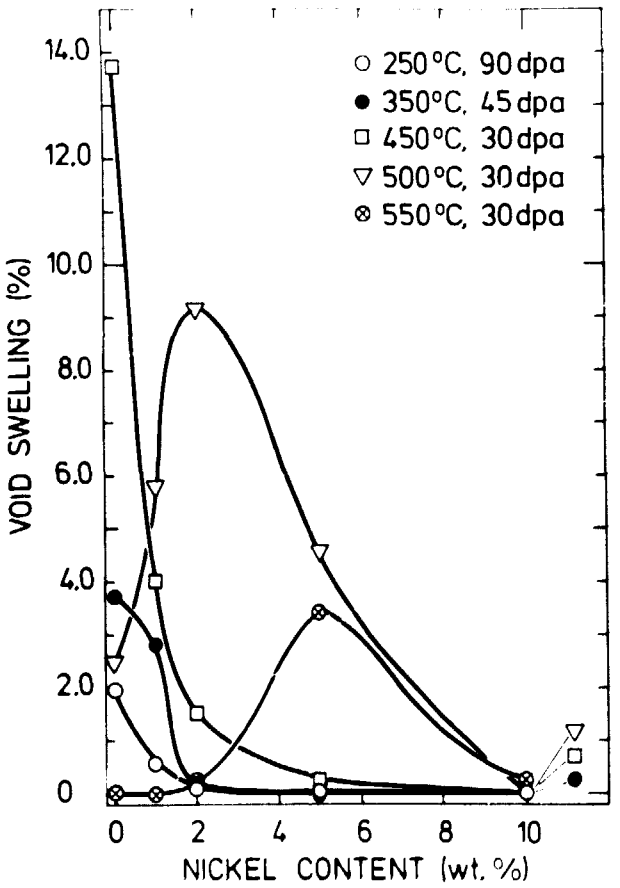

Fig. 4. Void swelling in Cu-Ni versus Ni content at various (HVEM) irradiation temperatures.

The study of void swelling in copper-nickel alloys (1-10 wt% Ni) was continued. The decrease in swelling with increasing Ni content is ascribed to the fact that Ni clusters act as recombination centres, because of the binding between Ni and vacancies or interstitials. The effect of injected helium on void swelling was investigated.

Blistering in Stainless Steel

Experiments were performed on the blistering in steels (type 316 and type 20Cr-20Ni) resulting from bombardment with helium ions. The irradiation was performed at accelerators at Culham and Harwell, UK. The effect of grain boundaries was studied by irradiation at room temperature. Very small helium bubbles were detected by transmission electron microscopy both at grain boundaries and in the interior of the grains.

Recrystallization in Dispersion-Hardened Metals

Experiments on recrystallization, i.e. nucleation and growth of nuclei, were performed on Al-Al_2O_3 (produced from pure Al). The experiments were supplemented by studies of grain growth after recrystallization. Dynamic studies of the nucleation in aluminium dispersion-hardened with Al_2O_3 particles were made in the high voltage electron microscope at AERE Harwell, UK.

Oxides and Hydrides

The investigation of non-stoichiometric cerium oxides was continued and extended to include plutonium oxides (in collaboration with the Transuranium Institute, Karlsruhe, Germany).

A preliminary study was made of the possibility of using metallic hydrides as storage and transport medium for hydrogen.

TECHNOLOGY AND MATERIALS DEVELOPMENT

Projects in this field concerned the development and examination of materials and techniques of relevance to nuclear energy or non-nuclear industrial applications.

Fibre-reinforced Plastics

Fabrication of plastics reinforced with glass fibres, with carbon fibres, or with a mixture of both types of fibres was investigated.

It was found that adhesive bonding of continuous carbon-fibre-reinforced plastics with lap joints required clean surfaces and produced joint efficiencies of 0.12. Bonding between reinforced plastics and wood or aluminium was also possible, with joint efficiencies of about 0.3 for wood and about 0.6 for aluminium. A sandwich beam design with skin layers of carbon-fibre-reinforced plastics was found to exploit the properties of these plastics in an efficient way.

It was found that ordinary high-speed steel drills wear out rather rapidly when used for machining of carbon-fibre-reinforced plastics, while drills of cemented carbides have fairly long lifetimes.

Corrosion Testing

Measurements of the corrosion rate in the secondary cooling system of the DR 3 test reactor by means of a Corrosometer probe indicated a metal loss from steel of about 0.08 mm per year. This corrosion rate is 2 to 3 times higher than that found by using immersed specimens. The rate is not very different from that usually found in untreated, air-saturated fresh water. This indicates that the corrosion inhibitors (non-chromates) now added to the cooling water only have a limited effect on the average corrosion rate.

Ultrasonic Soldering

It has been found that aluminium parts dipped in ultrasonically agitated solder baths are dissolved at a rate much higher than in conventional dip-brazing baths. In particular, thin aluminium wires are difficult to handle in an ultrasonic solder bath because they dissolve in a very short time - often less

than one second. To overcome this problem, a series of tests were started to correlate the dissolution rate of aluminium with the ultrasonic effect. The solder baths used are Sn-5Ag and Zn-5Al.

Welding of Thin Aluminium Plate

The characteristics of two different AC GTAW (Gas Tungsten Arc Welding) welding machines were investigated for a selected thin plate joint configuration, and optimal welding parameters were established. A permanent set-up with high-frequency filters and a two-channel oscilloscope was built in order to measure the welding current and voltage. Furthermore pulsed DC GTAW welding was tried on the same thin plate configuration. This process proved to produce a narrower weld for a given penetration and gives better reproducibility than the AC GTAW-method.

Joining Methods for Fuel Pins

A feasibility study was carried out on capacity discharge welding of zircaloy in a configuration suitable for third closures for PWR fuel pins. The preliminary evaluation based only on leak testing and metallography shows that the method is promising compared with the earlier used GTAW (Gas Tungsten Arc Welding) process.

Nickel-brazed Inconel

The investigation of the getter brazing process on Inconel X-750 was continued. Joints of Inconel X-750 were brazed with the use of filler metals AWS BNi6 and BNi7 and varying surface treatments. The joints were brazed either with the getter method at a temperature of 1025^{o}C and a pressure of 10^{-2} torr or with the normal high vacuum brazing technique at 1075^{o}C and 10^{-5} torr. The joints were tested for shear strength; the getter-brazed specimens had strengths equal to or better than those of the vacuum-brazed specimens. Earlier experiments showed that wetting properties also are better in getter-brazing than in vacuum-brazing. Therefore the use of zirconium getters in vacuum-brazing makes it possible to reduce the brazing temperature by 50^{o}C and increase the pressure from 10^{-5} to 10^{-2} torr. The latter pressure can be obtained with an ordinary mechanical pump.

FUEL ELEMENTS

The Danish fuel elements in the Kahl and Halden reactors continue to perform well and demonstrate the adequacy of the design and manufacturing processes.

The irradiation of UO_2-Zr fuel pins in the DR 3 reactor at Risø includes standard BWR and PWR type tests irradiated to very high burn-ups. A number of special tests such as power ramp tests are also being made.

Additional information on fuel performance will become available as a result of international collaboration arrangements, i.e. the OECD Halden Reactor Project (Norway), the "Interramp" (BWR fuel) and the proposed "Overramp" (PWR fuel) projects at Studsvik (Sweden), the information exchange with NRC (USA), and the EEC sponsored activities (Brussels) related to Pu recycling in LWRs.

Danish Fuel Element Irradiations in the Kahl and Halden Reactors

The four Danish fuel elements in the German BWR power reactor went on power for the first time in 1975. Irradiation was continued and these elements have now achieved an estimated average burn-up of 5,700 MWD/t UO_2. Two short test fuel pins, manufactured from the same UO_2 and Zr materials as the Kahl fuel pins, have now reached a burn-up of 21,700 MWD/t UO_2 in the DR 3 reactor.

Irradiation of the five test fuel elements in the Halden reactor (Norway) was continued. They have now reached the following burn-ups (average assembly):

IFA No.	148	161	165	201	202
MWD/t UO_2	32,300	37,500	32,800	23,300	19,600

The maximum local burn-up of 47,000 MWD/t UO_2 was achieved with IFA 161.

UO_2-Zr Irradiations at Risø

In the test fuel irradiation programme at DR 3, about 150 fuel pins have been or are being irradiated in some 90 tests.

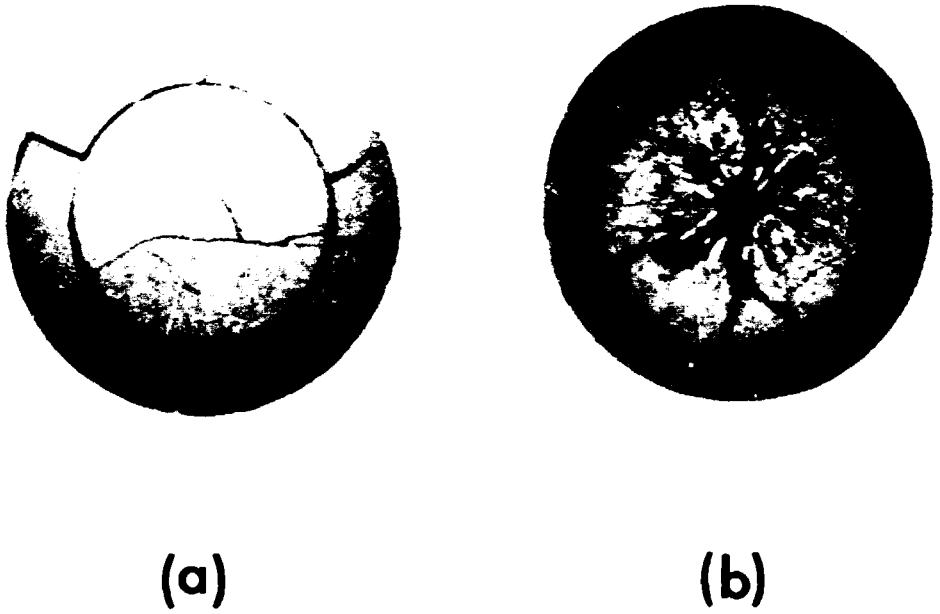

Fig. 5. Cross sections of LOWI (a) and reference fuel (b) irradiated 23 days at 650 W/cm. The LOWI temperature level has been so low that no grain growth has occurred, whereas the standard pellet exhibits columnar grain growth as well as a center void.

Standard fuel pins have reached maximum burn-up levels of 43,900 MWD/t UO_2 for BWR fuel and 32,200 MWD/t UO_2 for PWR fuel.

The series of power ramp experiments included two pellet pins and one vipac pin from an assembly previously irradiated in the Halden reactor to 20,600 MWD/t UO_2. In addition, a PWR pin was ramp-tested at 17,000 MWD/t UO_2. A number of test fuel pins with the special low-interaction ("LOWI") UO_2 pellet design was manufactured, and two irradiation experiments in DR 3 were initiated. One of these was unloaded after one reactor period (23 days) to obtain early performance results. Profilometry results for this test showed no signs of pellet/clad interaction for the LOWI pin, whereas there were signs of deformation in the cladding of the accompanying reference pin that contained pellets of standard design.

Computer Modelling of Fuel Pin Performance

A fast one-dimensional fuel-pin performance code, HOTCAKE/T, was developed for routine analysis based on any given power history. This code was used for analyses of Danish irradiation experiments. HOTCAKE/T was also used to predict the results of a series of ramp tests within the international "Interramp" project, and its performance compared favourably with that of the codes used by other project participants.

As a further development, a version of the code with ridging capability HOTCAKE/TR was produced.

Post-Irradiation Examination Methods

The new test bench for non-destructive measurements is now used routinely with automatic data processing and presentation for γ-scanning, γ-spectrometry, γ-densitometry, and profilometry of fuel pins.

Eddy-current equipment is being adapted to the bench, so that this type of observation may also be included in the computerized data presentation.

A facility for micro-γ-scanning was constructed.

NON-DESTRUCTIVE TESTING

The projects in this section deal with implementation of the inspection techniques that are necessary for experiments in nuclear fuel element technology and materials technology.

X-ray Control

The use of X-ray paper instead of films can considerably reduce the production time of X-ray pictures. Exposure curves for aluminium and steel were established for this technique. The interpretation of the X-ray paper pictures depends on the densitometric properties of the paper and work was started to obtain the necessary data.

Neutron Radiography

Neutron radiography has been adopted as a standard procedure for post-irradiation examination of fuel rods, and during the year a total of 800 pictures was produced.

The use of neutron radiographs for the metrology of fuel pins was investigated and it seems that the limit of accuracy is about 100 microns with the present film material. Newly developed film material from Kodak may permit an increase in accuracy.

Acoustic Emission (AE)

The use of the AE technique in industry is increasing, and problems of interest to Danish industry were investigated. As an example a procedure was established for the periodic inspection of intermediate-size pressure vessels made from a special HY steel. Here the AE technique is used to detect crack-growth during pressure testing; if crack-growth occurs, vessels may fail at operating pressures lower than the test pressure. A programme was started to investigate the nature of AE signals with tensile specimens as test objects.

Tube Inspection

(In collaboration with the Elsinore Shipyard)

A number of repeated dimensional tests on a set of tubes were made with the tube inspection system in order to improve the stability and accuracy of measurements. The tests were made under stationary and dynamic conditions and the instruments as well as the procedures were checked.

The influence of water temperature on the measurements was thoroughly investigated and it seems that, depending on the required accuracy, the problem can be solved by a temperature compensation unit or a temperature-controlled water system, or a combination of the two. In most cases a system with temperature-controlled water circulation would be adequate to meet the specifications.

PARTICIPATION IN INTERNATIONAL COLLABORATION

The department is engaged in the following types of international collaboration: joint technical projects, committee work, reception of research fellows, and technical and scientific meetings. Participation in the OECD reactor project at Halden was continued. Six Danish fuel elements are at the moment being tested under irradiation in the Halden reactor.

A joint technical project, irradiation in the DR 3 of zircaloy-clad uranium-dioxide/plutonium fuel rods, was continued (with the AB Atomenergi, Sweden). Work continued on the joint programme on examination of advanced zirconium alloys for water reactors (with the UKAEA, United Kingdom, AB Atomenergi, Sweden, IFA, Norway, and the Finnish AEC). The Department took part in a Scandinavian working group on hot cell techniques.

The department was represented on the following committees:

The Halden Programme Group
The IAEA Working Group on "Reliability of Reactor Pressure Components"
The "Interramp" Project Committee
The CEC-NEA Working Group "Material and Mechanical Problems Related to the Safety Aspects of Steel Components in Nuclear Plants"
The Working Group "Nuclear Corrosion" under the "European Federation of Corrosion"
The EEC Advisory Committees for Programme Management: "Plutonium and Transuranium Elements", "Solid State Physics", and "Plutonium Recycling in Thermal Reactors"
The Council of the International Confederation of Thermal Analysis
The Nordic Committee for Thermal Analysis

and in the following Technical Commissions of the International Institute of Welding:

Commission I "Gas Welding and Allied Processes", Subcommission A "Brazing and Surfacing"
Commission IX "Behaviour of Metals Subjected to Welding"
Commission X "Residual Stresses and Stress Relieving. Brittle Fracture"

PARTICIPATION IN THE INTERTOOL EXHIBITION

Risø participated with a stand of 300 m^2 in the INTERTOOL-76 exhibition which took place at the Bella Center in Copenhagen 23-27 November 1976. The Metallurgy Department contributed to the stand with posters and with working equipment. For instance, tensile testing with electronic data collection and processing was performed at the stand as a demonstration of the INSTRON-5 programme developed at the department. Also, the new tube inspection system - developed in collaboration with the Elsinore Shipyard, Ltd. - was demonstrated at the stand.

The posters illustrated various techniques, for instance the methods of ultrasonic soldering and vacuum brazing, both of which are more compatible with environmental considerations than conventional methods. The department's involvement in alternative energy research was illustrated by posters about fibre materials technology and hydrogen storage in metals.

The production of nuclear fuel elements for power reactors as well as test reactors was illustrated. This part of the exhibition was supplemented with two films shown at the stand. One film was about the production of uranium from Greenland ore, the other followed the fabrication of the fuel elements that the Metallurgy Department, in collaboration with the Elsinore Shipyard, Ltd., has produced for the German Kahl reactor.

In connection with the exhibition the Metallurgy Department arranged a meeting with invited representatives from Danish industry in the Bella Center on 26 November. Lectures on the following selected topics within the range of activities of the department were given at the meeting:

H. Lilholt: "Fibre Materials"
A. Nielsen: "Safety of Steel Constructions"
O. Toft Sørensen: "Thermoanalytical Methods"
K. Rørbo: "High Temperature Alloys"
A. Jensen: "Nuclear Fuel Elements"

Both the stand and the meeting were very well visited, and the exhibition resulted in contacts with a number of potential customers from the Danish industry.

EDUCATION AND TRAINING

N. Hansen and K. Rørbo gave regular lectures on materials science to students at the Danish Academy of Engineering. T. Leffers, H. Lilholt, K. Rørbo, and B.N. Singh lectured on physical material science to students at the Technical University of Denmark. N. Hansen, T. Leffers, and H. Lilholt acted as external examiners at examinations for the Technical University of Denmark.

Four scholarship holders, three from Egypt and one from India, worked in the Department on projects in physical metallurgy and in post irradiation examination.

Undergraduate Projects

Six students from the Department of Mechanical Engineering at the Danish Academy of Engineering worked in the Department on the following projects in preparation for their bachelors' theses:

Karsten Wikkelsø:	Grain Growth in Al-Al_2O_3 Alloys
Søren W. Hansen:	Recrystallization of Cu-Al and Cu-Al_2O_3 Alloys
Carl Kai Nielsen:	Adhesive Bonding of Carbon-Fibre Reinforced Plastic and other Materials
Henrik Burkal:	Constructional Elements in Carbon-Fibre Reinforced Plastic and other Materials
Erik M. Olsen:	Evaluation of the Sensitivity of Steel to Hydrogen Embrittlement
Gorm Hagensen:	Machining of Carbon-Fibre Reinforced Plastic

One student from the Department of Structural Properties of Materials at the Technical University of Denmark, K. Viggo Rasmussen, worked in the Department on the project "Irradiation Damage in Zr-Alloys Investigated by Electron Microscopy" in preparation for his master's thesis.

Post-graduate Projects

Four post-graduate students from the Technical University of Denmark worked in the Department on the following projects in preparation for their licentiate (Ph.D.) theses:

C.P. Debel:	Fracture Mechanics
P. Brøndsted:	Strengthening Mechanisms in Cu-Alloys
B.S. Andersen:	Simulation Models for Deformation Processes, Especially Deep Drawing
I. Misfeldt:	Probabilistic Fracture Mechanics Applied to Fuel Pods (in collaboration with the Reactor Department).

Degrees Conferred

The University of Lund, Sweden, conferred the degree of tekn.dr. on O. Toft Sørensen.

PUBLICATIONS

Metallurgy Department Progress Report for the period 1 January to 31 December 1975.
Risø Report No. 340 (1976) 38 pp.

The activities of the Metallurgy Department at Risø during 1975 are described. The work is presented in four chapters: General Materials Research, Technology and Materials Development, Fuel Elements, and Non-Destructive Testing. Furthermore, a survey is given of the department's participation in international collaboration and of its activities within education and training. A list (with abstracts) of publications and lectures by the staff during 1975 is included.

Structure of the $\langle 01\bar{1}0 \rangle$ Dislocation in Sapphire.
J.B. Bilde-Sørensen, A.R. Thölén, D.J. Gooch, and G.W. Groves, Phil. Mag. 33 (1976) 877-889.

Experimental evidence of the existence of $\langle 01\bar{1}0 \rangle$ dislocations in the $\{2\bar{1}\bar{1}0\}$ prism planes in sapphire has been obtained by transmission electron microscopy. By the weak-beam technique it has been shown that the $\langle 01\bar{1}0 \rangle$ dislocation may dissociate into three partials. The partials all have a Burgers vector of $\frac{1}{3}\langle 01\bar{1}0 \rangle$ and are separated by two identical faults. The distance between two partials is in the range 75-135 Å, corresponding to a fault energy of 320±60 mJ/m^2. Perfect $\langle 01\bar{1}0 \rangle$ dislocations have also been observed. These dislocations exhibited either one or two peaks when imaged in the $(03\bar{3}0)$ reflection by the weak-beam technique. The interpretation of the electron micrographs has been supported by computer simulation of the dislocation images. A faulted dipole has been observed, indicating that some of the prismatic loops often seen to lie in rows along [0001] are faulted with a $\frac{1}{3}\langle 01\bar{1}0 \rangle$ Burgers vector. A mechanism for the dipole formation is suggested.

Weak-Beam Electron Microscopy of Dislocations in Sapphire.
J.B. Bilde-Sørensen, A.R. Thölén, D.J. Gooch, and G.W. Groves, In: High Temperature Materials Phenomena, Proceedings of the 4th Nordic High Temperature Symposium, Helsinki, 13-15 June 1975. Vol. 2. Edited by M. Tilli (Dept. of Mining and Metallurgy, Helsinki University of Technology, Helsinki, 1975) 175-196.

The abstract appeared in the previous progress report p. 31.

Production of Tungsten Fibre Reinforced Nickel Composites by Liquid Infiltration.

H. Carlsen and H. Lilholt, In: High Temperature Materials Phenomena, Proceedings of the 4th Nordic High Temperature Symposium, Helsinki, 13-15 June 1975. Vol. 2. Edited by M. Tilli (Dept. of Mining and Metallurgy, Helsinki University of Technology, Helsinki, 1975) 67-76.

The abstract appeared in the previous progress report p. 31.

High Temperature Tensile and Creep Properties of Tungsten Fibre Reinforced Nickel Composites.

H. Carlsen and H. Lilholt, In: High Temperature Materials Phenomena, Proceedings of the 4th Nordic High Temperature Symposium, Helsinki, 13-15 June 1975. Vol. 2. Edited by M. Tilli (Dept. of Mining and Metallurgy, Helsinki University of Technology, Helsinki, 1975) 99-114.

The abstract appeared in the previous progress report p. 31.

Production of Tungsten Fibre Reinforced Nickel Composites by Liquid Infiltration and Sinter Forging.

H. Carlsen, H. Lilholt, and A. Kannappan, In: Metal Composite Material for High Temperatures. Final Report, Nordforsk Project Committee for Metallic Composites. (Nordforsk, Stockholm, 1976) 13-25.

Production of fibre reinforced metals by two techniques, liquid infiltration and sinter forging, was studied using continuous tungsten fibres and pure nickel matrix.

During liquid infiltration the mould was heated in a purified argon atmosphere. By high frequency induction heating, the coil was designed to preheat the fibres to a temperature lower than the melting point of the nickel. Specimens of 3 mm diameter up to 150 mm length and a fibre content up to 50 volume % were produced. The nickel matrix was polycrystalline and some recrystallization occurred in the surface zone of the heavily drawn fibres. The advantages of the method are: easy and flexible handling, and a short duration of the casting operation; the disadvantages regarding the nickel-tungsten system being dissolution and recrystallization of the fibres.

Sinter forging of regular wire arrays embedded in a powder matrix, is an alternative production process. Tungsten fibres wound on a frame were placed in a trough and nickel powder was vibrated into the trough. The whole assembly was presintered for 1 hour at 900°C in hydrogen, producing a compact of sufficient strength to keep the wires straight when the frame was removed. After trimming off the wire ends, this compact was heated to 1100°C in hydrogen and forged in a closed die to full density. In this way, bars of dimensions 4 × 10 × 100 mm with up to 23 vol.% fibres, were made. The ehat treatment during manufacture produced no detectable recrystallization or other matrix-fibre interactions.

High Temperature Tensile and Creep Properties of Tungsten Fibre Reinforced Nickel Composites.

H. Carlsen and H. Lilholt, In: Metal Composite Material for High Temperatures. Final Report, Nordforsk Project Committee for Metallic Composites. (Nordforsk, Stockholm, 1976) 61-76.

Specimens consisting of continuous tungsten fibres in a nickel matrix were made by liquid infiltration and by powder-forging. Tensile testing showed that the cast composites were rather brittle below 300-400°C, and had low and scattered tensile strengths compared to powder-forged composites. Above this temperature range the two types of composites behaved similarly. Creep properties of cast specimens were investigated at 800 and 900°C. The results showed the expected dependence of creep rate on tensile stress, but the absolute creep strengths were low compared to a simple model of a composite with creeping, continuous fibres.

Accuracy of Dimension Measurements from Neutron Radiographs of Nuclear Fuel Pins.

J.C. Domanus, Risø-M-1860 (1976) 8 pp.

A review is given of different methods used for dimension measurements from neutron radiographs and results are presented of an investigation performed, using unirradiated fuel pins with calibrated UO_2 pellet diameters and fuel-to-clad gaps.

Projection microscope, three types of travelling microdensitometers and an electronic image analyzer were used to measure diameters and gaps from neutron radiographs produced at Risø DR 1 and Studsvik (Sweden) R2 reactors, using different brands of X-ray films and transfer technique with 0.1 mm Dy foil.

Application of Radiographic Paper to Quality Control of MTR Fuel Elements.

J.C. Domanus, In: Nuclear Fuel Quality Assurance. Proceedings of a Seminar held by the International Atomic Energy Agency in Oslo, 24-27 May 1976 (IAEA, Vienna, 1976) 349-359. (Also as Risø-M-1861 (1976) 13 pp.)

To prove that X-ray paper is of adequate quality for use in the radiological quality control of Material Testing Reactor (MTR) fuel elements, a comparison was made between X-ray paper and X-ray film. Radiological control is applied in radiography of U/Al cast blocks (30-mm thick) from which MTR fuel plates are rolled down. The plates themselves are also controlled by radiography, and fuel plates radiographs can be further used to assess the homogeneity of the uranium distribution in the plate. The quality of plate radiographs was assessed by measuring radiographic density contrast under an Al step wedge. The Al equivalents of a test plate were also determined by densitometric scanning. The quality of U/Al cast blocks radiographs was judged by using Image Quality Indicators (IQIs) of the ISO type (in the form of holes drilled in the block itself) and of the ASTM type (plate with holes, placed on the block). All comparative work was done using Kodak and Agfa-Gevaert X-ray paper and fluorescent intensifying screens as well as X-ray film (no screen for plate radiography, Pb intensifying screens for block radiography). The results proved that X-ray paper is of sufficiently good quality to be used for radiological control of MTR fuel elements. Its use gives considerable gains in exposure and processing times, as well as in the cost of the radiographic material.

Radioisotopes in Non-Destructive Testing.

J.C. Domanus, Risø-M-1906 (1976) 123 pp.

After defining NDT and comparing this concept with destructive testing, a short description is given of NDT methods other than radiologic. Basic concepts of radiologic methods are discussed and principles of radiography are explained. Radiation sources and gamma radiography machines are next reviewed and radiographic inspection of weldings and castings is described. A brief description is given of the radiographic darkroom and accessories. Other radioisotope methods, such as neutron radiography and radiometry are shortly reviewed. The presentation is concluded by cost estimations for the radioisotopic equipment.

Studies of Dislocations Using Two-Dimensional Model Systems.

E.J. Jensen and P.O. Esbjørn, In: Proceedings of the 1976 International Conference on Computer Simulation for Materials Applications, Gaithersburg, Maryland, 19-21 April 1976. Edited by R.J. Arsenault, J.R. Beeler, and J.A. Simmons. (Nuclear Metallurgy, vol. 20, part 1). (National Bureau of Standards, Gaithersburg, 1976) 459-470.

Properties of dislocations in a two-dimensional Lennard-Jones model solid are investigated. Using linear elasticity theory, expressions for the dislocation energy and entropy are derived analytically and related to results of molecular dynamics calculations on the model. Resulting configurations are compared with bubble raft dislocations and with results of earlier computer calculations.

Computer Studies of Dislocation Properties Using Two-Dimensional Model Systems.

P.O. Esbjørn and E.J. Jensen, J. Phys. Chem. Solids 37 (1976) 1081-1091.

The physical properties of dislocations are investigated by means of a two-dimensional Lennard-Jones solid. Using a selfconsistent linear theory for the elastic properties of two-dimensional systems, analytic expressions for the energy and entropy of a dislocation in the center of a circular finite model are derived. The energy and entropy formulas are compared with the results of molecular dynamics calculations on the model. The resulting atomic structure of the dislocation core is compared with the structure of bubble raft dislocations and with the results of earlier computer calculations.

Experience with a New Ultrasonic Inspection System for Non-Destructive Examination of Canning Tubes.

H.E. Gundtoft, T. Nielsen, and C.C. Agerup, In: Nuclear Fuel Quality Assurance. Proceedings of a seminar held by the International Atomic Energy Agency in Oslo, 24-27 May 1976 (IAEA, Vienna, 1976) 265-279.

(Also as Risø-M-1850 (1976) 15 pp.)

The Risø/HV Tube Inspection System is designed for fast automatic inspection of precision tubes for dimensions and defects. The system is built around a rotating water chamber with eight ultrasonic transducers. The tube handling and evaluation of the results could be made in different ways by various combination of modules in the system. By standard inspection of canning tubes for nuclear fuel, the inspection speed is 6 m of tube per minute with the chamber rotating 3000 rev/min; tubes are fed through the system without rotation, and, owing to the conical tube guide used, very gentle treatment of the tubes is assured. Evaluation and development work on this system has been performed during the last three years and has been based on practical experience with tube inspection. The accuracy and stability of the system has been improved by innovation in both the mechanical system (especially water circulation and temperature regulation) and the electronic modules (ultrasonic equipment). Also the ultrasonic transducers have been studied and changed. The calibration procedure has been simplified and improved both for dimensions and defects. The influence of the different improvements on stability and accuracy during actual tube inspection has been ascertained by repeated inspection of the same 17 tubes. The results from these inspections indicate an accuracy and stability of less than 5 μm on dimension measurements during actual tube examinations.

Computer Control in Non-Destructive Testing.

H.E. Gundtoft and N. Nielsen, Risø-M-1868 (1976) 8 pp.

In Risø's automatic tube inspection system, data (more than half a million per tube) from ultrasonic dimension measurements and defect inspections are fed into a computer that simultaneously calculates and evaluates the results.

The Effect of Grain Size on the Work-Hardening of Aluminium at Room Temperature.

N. Hansen, In: Grain Boundaries, Proceedings of the Institution of Metallurgists Spring Residential Conference, Jersey, 9-12 April 1976. (The Institution of Metallurgists, London, 1976) F8-F12.

Tensile stress/strain data over a strain range from 0.2 to 30% were obtained at room temperature for 99.999 and 99.5% aluminium as a function of grain size.

The yield-stress/grain-size relationship can be expressed by a Petch-Hall relation with approximately the same slope for the two materials. The flow-stress/grain-size relationship can adequately be expressed by a modified Petch-Hall relation; for 99.999% aluminium material the slope increases with strain through a maximum around 15-20%, whereas for 99.5% aluminium the slope decreases with the strain to zero at strains about 10%.

The flow-stress/grain-size relationship was analyzed in terms of matrix strengthening and grain boundary strengthening according to the dislocation concept of Ashby. At intermediate strains this approach gives a good description of the effect of strain, grain size and purity on the flow stress.

A Review of the Effect of Neutron Irradiation on the Deformation Behaviour of Copper and Copper Alloys.

H.R. Higgy, Risø-M-1871 (1976) 31 pp.

The basic mechanisms of irradiation hardening are described. The effects of neutron dose, alloying and pre-irradiation deformation on the deformation behaviour of neutron-irradiated copper and its alloys are considered. The discrepancy in the reported data is discussed. Substitutional and interstitial additions are found to influence the irradiation hardening rate, while pre-irradiation deformation has no influence. The deformation behaviour of copper is found to alter as a result of irradiation and alloying.

Industrien betaler for uvisheden omkring valg af energi. (The Industry Pays for the Uncertainty Concerning Choice of Energy Sources).

A. Jensen, Dagbladet Børsen 21 September 1976.

The condition for Danish industrial participation in the construction of nuclear power plants in Denmark is that the industry to a certain extent possesses the necessary know-how. A period of uncertainty whether to use this nuclear energy or not will therefore either impose extra costs on the industry to maintain the level of know-how or lead to less participation in the construction when a decision is made.

Analysis of Overpower Performance of High-Burnup Pellet and Vipac UO_2-Zr Fuel Pins.

P. Knudsen, C. Bagger, and N. Kjær-Pedersen, Trans. Am. Nucl. Soc. 24 (1976) 172.

A pellet and a vipac UO_2-Zr fuel pin were irradiated in a test assembly to 20,600 MWD/te UO_2 (avg. ass'y). Subsequent ramp testing of the individual fuel pins to max. heat loads of 720 and 640 W/cm, respectively, caused failure in both cases.

The experiments were analyzed by Danish fuel performance codes. The analyses were in reasonable agreement with the experimental results insofar as the local clad stresses and strains were of sufficient magnitude to cause failure in connection with the assumed action of a stress corrosion mechanism.

Irradiation Induced Dislocation Climb Sources.

T. Leffers and P. Barlow, In: Microscopie électronique a Haute Tension. Textes des Communications présentées au 4e Congres International, Toulouse, 1-4 Septembre 1975. Édité par B. Jouffrey and P. Favard. (Société Française de Microscopie Electronique, Paris, 1976) 201-204.

The abstract appeared in the previous progress report p. 33.

Mechanical and Physical Properties: Rapporteur's Report.

H. Lilholt, In: Proceedings of the 1975 International Conference on Composite Materials, Geneva 7-11 April, and Boston, 14-18 April 1975. Vol. 1. Edited by E. Scala, E. Anderson, I. Toth, and B.R. Noton. (The Metallurgical Society of AIME, New York, 1976) 723-741.

The abstract appeared in the previous progress report p. 34.

A Study of Nickel Reinforced with Tungsten Wires, a Potential High Temperature Material.

H. Lilholt and H. Carlsen, In: Proceedings of the 1975 International Conference on Composite Materials, Geneva, 7-11 April, and Boston, 14-18 April 1975. Vol. 2. Edited by E. Scala, E. Anderson, I. Toth, and B.R. Noton. (The Metallurgical Society of AIME, New York, 1976) 1321-1333.

The abstract appeared in the previous progress report p. 34.

Application of Hastelloy X in Gas-Cooled Reactor Systems.

C.R. Brinkman, P.L. Rittenhouse, W.R. Corwin, J.P. Strizak, and A. Lystrup, ORNL/TM-5405 (1976) 59 pp.

Hastelloy X, an Ni-Cr-Fe-Mo alloy, may be an important structural alloy for components of gas-cooled reactor systems. Expected applications of this alloy in the High-Temperature Gas-Cooled Reactor (HTGR) are discussed, and the development of interim mechanical properties and supporting data are reported. Properties of concern include tensile, creep, creep-rupture, fatigue, creep-fatigue interaction, subcritical crack growth, thermal stability, and the influence of helium environments with controlled amounts of impurities on these properties. In order to develop these properties in helium environments that are expected to be prototypic of HTGR operating conditions, it was necessary to construct special environmental test systems. Details of construction and operating parameters are described. Interim results from tests designed to determine the above properties are presented. To date a fairly extensive amount of information has been generated on this material at Oak Ridge National Laboratory and elsewhere concerning behavior in air, which is reviewed. However, only limited data are available from tests conducted in helium. Comparisons of the fatigue and subcritical growth behavior in air between Hastelloy X and a number of other structural alloys are given.

Ultrasonic Soldering Horn Lifetime.

A. Lystrup, Weld. J. 55 (1976) 109.

In some ultrasonic soldering equipment two inclined horns reach down into the solder bath from above. The length of an unused horn is 132.5 mm, but during operation the end of the horn erodes and the ultrasonic output effect decreases. Normally acceptable soldered joints are produced if the horn is not eroded more than 6 mm. If the horn is longer than 132.5 mm, the generator has difficulties in starting the oscillation of the horn. However, if a starting impulse is provided by another ultrasonic horn, the vibrations can be initiated at any time, even if the horn is 10 mm longer than normal. In addition the output effect from the longer horn is much larger than that from the shorter one. This means that both the quality of the joints and the lifetime of the horn can be increased. By choosing the right length of the horn one is able to increase the lifetime at least 3 times.

Measurement of the Ultrasonic Effect in an Ultrasonic Solder Bath.

A. Lystrup, Weld. J. 55 (1976) 309s-313s.

In ultrasonic soldering ultrasonic effect is transmitted to a solder bath by ultrasonic horns. During operation the horns erode and the transmitted effect becomes less and less. Thus, to obtain good process control it is necessary to be able to measure the ultrasonic effect, i.e., sound level, in the solder bath. This paper describes a recently developed method of measuring this effect. The measurements are carried out with an ultrasonic crystal and the magnitude of the effect is registered on a voltmeter. Just as a piezoelectric crystal is able to send out mechanical vibrations when subjected to an ac voltage, it is able to emit an ac voltage when mechanically vibrated. The measurements show that the ultrasonic effect depends very strongly on the length of the transmitting ultrasonic horn, the chemical composition of the solder, and the distance from the transmitting horn and, further, that the effect is independent of the temperature of the solder bath. During the work, a method to produce ultrasonic horns with long lifetime was found.

Instrumented Impact Testing as a Way to Obtain Further Information on the Behaviour of Steel in Welded Constructions.

A. Nielsen, Risø-M-1865 (1976) 18 pp.

The abstract appeared in the previous progress report p. 35.

Svejseproces for brændselselementer på Risø. (Welding Procedure for Fuel Elements at Risø).

P. Dreves Nielsen and J. Olsson, Svejsning 3 [5] (1976) 124-128.

The welding equipment and the procedure for welding of fuel elements are described. The radiographic control of the welds is discussed.

Steady-State Creep of Copper-Tungsten Fibre Composites.
O. Bøcker Pedersen, In: Proceedings of the 1975 International Conference on Composite Materials, Geneva, 7-11 April, and Boston, 14-18 April 1975. Vol. 1. Edited by E. Scala, E. Anderson, I. Toth, and B.R. Noton. (The Metallurgical Society of AIME, New York, 1976) 918-929.

The abstract appeared in the previous progress report p. 35.

The Dislocation Formation Volume in Ice.
R.M.J. Cotterill and O. Bøcker Pedersen, In: Proceedings of the 1976 International Conference on Computer Simulation for Materials Applications, Gaithersburg, Maryland, 19-21 April 1976. Edited by R.J. Arsenault, J.R. Beeler, and J.A. Simmons. (Nuclear Metallurgy, vol. 20, part 1). (National Bureau of Standards, Gaithersburg, 1976) 572-581.

Relaxations around a shear dislocation loop on a basal plane in ice have been studies by molecular dynamics. The model intermolecular potential included directional components which stabilized the open ice structure. A random phase approximation was introduced to simulate the disordered arrangement of hydrogen bonds. The dislocation formation volume was found to be zero within the limits of computational error.

The Bauschinger Effect in Single Phase Crystals.
O. Bøcker Pedersen, Dissertation, University of Cambridge (1976) 62 pp.

The theory for the transformation problem is briefly described. The mean stress is derived on the basis of a generalised version of the PMS model assuming elastic homogeneity. The derivation is carried out in terms of stress and in terms of free energy and the equivalence of the two derivations is demonstrated. The range of validity of the PMS model is discussed, and the model is evaluated in the situation of single slip for homogeneously and isotropically elastic materials. The calibration of the Bauschinger experiment for measuring internal stresses is described. The temperature change experiment is described. The present experiments are reported and results are given for forward resolved shear strains ranging from 0.1 to 0.6 for single crystals at 77°K and 293°K and for polycrystals at 293°K. It is shown that the correlation for two phase materials does not hold for pure copper. This result is given a qualitative interpretation on the basis of the picture of the dislocation structure suggested by electron microscopy and computer simulation. An effective accomodation factor is considered for dislocation tangles acting as obstacles to primary slip. Suggestions for further experiments are made.

Void Shrinkage in Stainless Steel During High Energy Electron Irradiation.

B.N. Singh and A.J.E. Foreman, Risø-M-1845 (1976) 23 pp.

During irradiation of thin foils of an austenitic stainless steel in a high voltage electron microscope, steadily growing voids have been observed to suddenly shrink and disappear at the irradiation temperature of 650°C; the phenomenon has been observed in specimens both with and without implanted helium. Possible mechanisms for void shrinkage during irradiation are considered. It is suggested that the dislocation-pipe-diffusion of vacancies from or of self-interstitial atoms to the voids can explain the shrinkage behaviour of voids observed during our experiments.

A Positron Annihilation Investigation into the Annealing of Copper after Neutron Irradiation at 50°C.

J.H. Evans, O. Mogensen, M. Eldrup, and B.N. Singh, In: Preprints of the Fourth International Conference on Positron Annihilation, Helsingør, 23-26 August 1976. Part 2. Paper No. E14. 71-75.

Angular correlation and positron lifetime measurements have been made during the isochronal annealing of copper following neutron irradiation at 50°C. Marked changes in positron annihilation parameters took place in the irradiation; these changes annealed out in a sharp recovery stage between ∿ 300 and 400°C, in close agreement with the work of Gauster and Mantl. It is suggested that the positrons could be detecting sub-microscopic voids formed in the irradiation which eventually shrink during the thermal annealing. It is argued that provided vacancies are mobile in the irradiation (which was above stage III) then void formation can take place by the normal preference mechanism. However, it is recognised that because of the complexities of the as-irradiated structure, with electron microscopy showing a high density of vacancy loops, interstitial loops and dislocations (but no visible voids), a less simple interpretation of the positron data in terms of these defects might be necessary - particularly if no supporting evidence for the void model can be obtained.

Acoustic Emission from Microcracks During Sliding Contact.

W.E. Swindlehurst and T.R. Wilshaw, J. Mat. Sci. 11 (1976) 1183-1186.

Acoustic emission generated by microcracks formed during sliding contact has been recorded. The study examines the relationship between the size and density of the microcracks and the recorded emission in detail. A significant reduction in emission strength with increasing microcrack density is observed and a physical explanation is provided.

Acoustic Emission in Brittle Solids.

W.E. Swindlehurst and T.R. Wilshaw, J. Mat. Sci. 11 (1976) 1653-1660.

A signal/source correlation study of the stress waves emitted during unstable microscopic Hertzian fracture in glass is described. A theoretical analysis of the variation in excess strain energy with applied load is made and the results compared with the experimental data covering a wide range of crack sizes.

High Temperature Studies of Thermodynamic Properties and Structures of Non-Stoichiometric Cerium Dioxides.

O. Toft Sørensen, In: High Temperature Materials Phenomena, Proceedings of the 4th Nordic High Temperature Symposium, Helsinki, 13-15 June 1975. Vol. 1. Edited by M. Tilli. (Dept. of Mining and Metallurgy, Helsinki University of Technology, Helsinki, 1975) 75-96.

The abstract appeared in the previous progress report p. 36.

Thermodynamic Studies at Higher Temperatures of the Phase Relationships of Substoichiometric Plutonium and Uranium/Plutonium Oxides.

O. Toft Sørensen, In: Plutonium 1975 and Other Actinides. Proceedings of the 5th International Conference on Plutonium and Other Actinides, Baden-Baden, 10-13 September 1975. Edited by H. Blank and R. Lindner. (North-Holland Publishing Company, Amsterdam, 1976) 123-131.

The abstract appeared in the previous progress report p. 36.

Thermodynamic Studies of Non-Stoichiometric Oxides by Thermoanalytical Methods.

O. Toft Sørensen, In: Proceedings of the First European Symposium on Thermal Analysis, Salford, UK, 20-24 September 1976. Edited by D. Dollimore. (Heyden and Sons Ltd., London, 1976) 109-110.

Recent thermogravimetric studies on non-stoichiometric cerium oxides, plutonium oxides and mixed uranium/plutonium oxides are reviewed in the paper. These studies have shown that, contrary to previous ideas, the non-stoichiometric phase-range for these oxides can be divided into subregions, some of which are still non-stoichiometric with a characteristic type of defect, whereas others definitely consist of a whole series of intermediate discrete compounds. Some of the conclusions drawn from the thermogravimetric studies have been verified by high-resolution electron-microscopy, this is also briefly discussed in the paper.

Rare Earth and Actinide Oxides. Thermodynamic and Electron Microscopy Studies.

O. Toft Sørensen, Risø Report No. 331 (1975). Thesis. 75 pp.

Partial molar thermodynamic quantities for oxygen in sub-stoichiometric cerium oxides (CeO_{2-x}), plutonium oxides (PuO_{2-x}) and mixed uranium-plutonium oxides ($(U,Pu)O_{2-x}$) were determined by thermogravimetric analysis in atmospheres of controlled oxygen pressures (CO_2/CO mixtures) in the temperature range 900-1450°C. Detailed analysis of the data obtained showed that the non-stoichiometric phase ranges for the three oxide-systems, which were previously described as a single, grossly non-stoichiometric phase, can be divided into several subregions each consisting of an apparent non-stoichiometric single phase. The finer details of the thermodynamic data, however, suggest that some of these subregions can be further split into ordered intermediate phases with compositions following the series M_nO_{2n-2}.

In order to verify some of the thermodynamic findings, supplementary high-temperature X-ray diffraction studies were made on CeO_2 at temperatures up to 855°C. At the higher temperatures between 790 and 855°C, a new phase of low symmetry was obtained. Indexing the powder pattern for this phase showed it to be isostructural with Pr_6O_{11} and with a monoclinic unit cell with a = 6.781±0.006 Å, b = 11.893±0.009 Å, c = 15.823±0.015 Å and β = 125.04±0.04°. The Ce_6O_{11} phase observed in the X-ray studies corresponds to one of the intermediate phases inferred from the thermodynamic data.

Supplementary high resolution electron microscopy studies were also conducted on reduced single crystals of CeO_2. On some particles reduced by beam heating in the microscope, a lamellae structure was observed and a model involving crystallographic shearing is proposed to explain this observation. In other beam-heated particles diffraction patterns were observed corresponding to the monoclinic superstructure found in the high-temperature X-ray studies. Finally patterns on particles reduced by a heat treatment in vacuum under well defined conditions showed that twinning can also take place in this oxide system.

Studies of Non-Stoichiometric Oxides by Thermoanalytical Methods.
O. Toft Sørensen, Thermochim. Acta 15 (1976) 227-237.

The abstract appeared in the previous progress report p. 37.

Thermodynamic Studies of the Phase Relationships of Non-Stoichiometric Cerium Oxides at Higher Temperatures.
O. Toft Sørensen, J. Solid State Chem. 18 (1976) 217-233.

Partial molar thermodynamic quantities for oxygen in nonstoichiometric cerium oxides were determined by thermogravimetric analysis in CO/CO_2 mixtures in the temperature range 900-1400°C. Under these conditions compositions within the range $2.00 \geq O/M \geq \sim 1.75$ could be obtained. A detailed analysis of the data shows that the α'-phase region in the phase diagram, previously described as a grossly nonstoichiometric phase, can be divided into several subregions each consisting of an apparent nonstoichiometric single phase. The finer details of the thermodynamic data, however, suggest that some of these subregions can be further split into ordered intermediate phases with compositions following the series M_nO_{2n-2}.

Supplementary high-temperature X-ray diffraction studies under vacuum were made at temperatures up to 855°C. At the higher temperatures between 790 and 855°C, a new phase of low symmetry was obtained. Indexing of the powder pattern for this phase showed it to be isostructural with Pr_6O_{11} and with a monoclinic unit cell with a = 6.781±0.006 Å, b = 11.893±0.009 Å, c = 15.823 ±0.015 Å, and β = 125.04±0.04°.

Alternativ Energiforskning (Alternative Energy Research).
C.U. Linderstrøm-Lang, C.J. Christensen, H. Neltrup, B. Vigeholm, S. Boisen Fritz-Rasmussen, and I. Rasmussen, Risø-M-1834 (1976) 34 pp.

A review of the possibilities on a short view for Risø to contribute to energy-oriented research and development in areas other than those covered by the present activities.

LECTURES AND CONFERENCE CONTRIBUTIONS

Kvalitetssikring - Eksterne og interne krav til dokumentation af kvalitetsstyringen. (Quality Assurance - External and Internal Demands to Documentation of the Quality Control).

C.C. Agerup, presented at the Conference on Quality Technique 1976, Jydsk Teknologisk Institut, Århus, October 1976. (Transcript available, 8 pp.)

Any buyer of industrial products expects to receive products of the specified quality. Experience has shown, however, that quality does not always comply with specification. This fact has caused customers to perform various quality assurance activities in order to avoid acceptance of defective products.

A New LWR Fuel Design.

J. Aukdal and A. Jensen, presented at the Enlarged Halden Programme Group Meeting, Sanderstølen, March 1976. (Transcript available, 2 pp.)

As an attempt to solve the PCMI-problem in LWR fuels a new design has been developed. The design change from that used at present is very small so that fabrication technology can be maintained. In the new design the traditional fuel pellet is split up in two parts: an outer ring, which bears all the enriched material, and an inner pellet made from natural uranium.

Accuracy of Dimension Measurements from Neutron Radiographs of Nuclear Fuel Pins.

J.C. Domanus, presented at the 8th World Conference on Non-Destructive Testing, Cannes, September 1976. (Available as Risø-M-1860).

Radioisotopes in Non-Destructive Testing.

J.C. Domanus, presented at the IAEA Advisory Group Meeting on Industrial Radioisotope Application with Particular Reference to Industries in Asia and the Far East, Bombay, December 1976. (Available as Risø-M-1906).

Computer Control in Non-Destructive Testing.

H.E. Gundtoft and N. Nielsen, presented at the 8th World Conference on Non-Destructive Testing, Cannes, September 1976. (Available as Risø-M-1868).

Risø Power Ramp Experience.

N. Hansen, presented at the 4th Water Reactor Safety Research Information Meeting, Washington, September 1976. (Proceedings to be published).

A review paper. A general description of irradiation and post-irradiation facilities at Risø is given. Projects within the fields of ramp tests, post-irradiation examination of pellet-clad interaction failures, and modelling of pellet-clad interaction failures are discussed. A new low-interaction fuel pellet design is escribed.

Fabrikation af brændselselementer. (Fabrication of Fuel Elements).

A. Jensen, presented to Dansk Metallurgisk Selskab, Lundtofte, May 1976. (Not available).

A description of methods and processes involved in the fabrication of nuclear fuel elements.

A New Concept for the Three-Dimensional Modelling of a Cracked Fuel Pellet.

N. Kjær-Pedersen, presented at the Enlarged Halden Programme Group Meeting, Sanderstølen, March 1976. (Transcript available, 9 pp.)

During power operation of pelletized UO_2 fuel pins the fuel cracks in a complicated way. This fact is the major obstacle to rigorous mathematical modelling of fuel pin behaviour. This paper presents a new set of concepts, which forms the basis for a reasonably fast computer model to account for transversal as well as radial fuel cracking. The concepts have been used in a disc model of a fuel pin. This model is briefly described and some calculational results are given. It is concluded that fuel cracking accounts for 20-25 percent of total radial pellet expansion. Ways of extending the disc model into a three-dimensional model that will account for pellet hourglassing and clad ridging are proposed.

Analysis of Overpower Performance of High-Burnup Pellet and Vipac UO_2-Zr Fuel Pins.

P. Knudsen, C. Bagger, and N. Kjær-Pedersen, presented at the American Nuclear Society Winter Meeting, Washington DC, November 1976. (Manuscript published in Trans ANS).

Power Ramp Performance of High-Burnup Vipac and Pellet UO_2-Zr Fuel Pins.

P. Knudsen and C. Bagger, presented at the Enlarged Halden Programme Group Meeting, Sanderstølen, March 1976. (Not available).

The Danish fuel assembly IFA 164 with vipac and pellet UO_2-Zr fuel pins was irradiated in HBWR to a burnup of 20,600 MWD/t UO_2 (avg. assembly). After non-destructive characterization at Risø, a vipac and a pellet fuel pin were overpower tested in the Danish reactor DR 3. The paper presents design and irradiation details as well as initial hot-cell results for these two ramp tests.

Driftserfaringer med brændselselementer (Operating Experience with Fuel Elements).

P. Knudsen, presented to Dansk Metallurgisk Selskab, Lundtofte, May 1976. (Not available).

The fuel in most commercial power reactors is Zircaloy-clad uranium dioxides as water-cooled rod bundles. The operating conditions for such fuel is summarized, and the various causes for operational disturbances in commercial reactors are reviewed.

Rectangular a<100> Loops in Electron Irradiated Cu-Ni. T. Leffers, J.B. Bilde-Sørensen, and P. Barlow, presented at the Joint Meeting of the Scandinavian Society for Electron Microscopy and the Nordic Society for Cell Biology 1976, Lyngby, June 1976. (Manuscript to be published in Scand. J. Met.).

Rectangular dislocation loops with Burgers vector a<100> are formed in Cu-Ni alloys during 1 MeV electron irradiation at 350-400°C in a high voltage electron microscope. The loops are sessile, pure edge in character with <110> line vectors, and of interstitial type. They may spontaneously split into two glissile prismatic $\frac{a}{2}$ <110> loops according to the reaction

$$a\,[100] \rightarrow \frac{a}{2}\,[110] + \frac{a}{2}\,[1\bar{1}0]$$

The reason for the relatively good stability of the a<100> loops probably is that they are dissociated according to the equations

$$a\,[100] \rightarrow \frac{a}{6}\,[2\bar{1}\bar{1}] + \frac{a}{6}\,[211] + \frac{a}{3}\,[100]$$

and $$a\,[100] \rightarrow \frac{a}{6}\,[2\bar{1}1] + \frac{a}{6}\,[21\bar{1}] + \frac{a}{3}\,[100]$$

Preliminary support for this dissociation has been obtained by weak-beam electron microscopy. The rectangular loops are emitted from dislocation climb sources. It is suggested that the source points consist of clusters of Ni atoms on {100} planes. The source points are not connected to any preexisting dislocation structure.

Defect Accumulation in Cu-Ni Alloys During HVEM Irradiation. P. Barlow, T. Leffers, and B.N. Singh, poster presented at the conference "Vacancies 76: Point Defect Behaviour and Diffusional Processes", Bristol, September 1976. (Not available).

The numerical results obtained in the void experiments and the loop growth experiments in Cu-Ni were presented. The void size and distribution and the dislocation behaviour in thicker and thinner foils (formation of "black dots" and dislocation sources, respectively) was illustrated by selected electron micrographs.

Mechanical Behaviour of Composites: Basic Approach: Creep Behaviour of Composites.

H. Lilholt, presented at the conference "Advances in Composite Materials", Euratom Joint Research Centre, Ispra, October 1976. (Transcript available, 28 pp.)

A review is given of creep theories for composites and their relation to the creep of the matrix; experiments are used to illustrate the theories.

Measurement of the Ultrasonic Effect in an Ultrasonic Solder Bath.

A. Lystrup, presented at the 5th American Welding Society International Soldering Conference, St. Louis, Missouri, May 1976. (Manuscript published in Weld. J.)

High-Resolution Electron Microscopy Studies of Fluorite-Related Cerium Oxides.

O. Toft Sørensen, presented at the 8th International Symposium on the Reactivity of Solids, Gøteborg, June 1976. (Proceedings to be published).

Previously, non-stoichiometric ceria, CeO_{2-x}, were considered to be a single grossly non-stoichiometric phase extending over a considerable composition range at higher temperatures. Recently, however, thermogravimetric measurements in atmospheres of controlled oxygen pressures have shown that the non-stoichiometric phase range can be divided into several subregions, some of which consist of an apparent single non-stoichiometric phase with a characteristic defect structure, whereas other regions apparently consist of a whole series of ordered intermediate phases. In order to confirm the existence of such ordered phases in the non-stoichiometric phase range, high-temperature X-ray studies as well as high-resolution electron microscopy studies on reduced single crystals of ceria, have been started; the results obtained so far will be discussed in this paper. As a basis for the discussion, the results of the thermogravimetric experiments will also be reviewed.

Thermodynamic Properties and Structures of Non-Stoichiometric Rare Earth- and Actinide Oxides.

O. Toft Sørensen, presented at the Arrhenius Laboratory, Stockholm, May 1976, and at the H.C. Ørsted Institute, Copenhagen, December 1976. (Not available).

Some recent thermodynamic data obtained by thermogravimetric measurements in atmospheres of controlled oxygen pressure on non-stoichiometric cerium dioxides (CeO_{2-x}), plutonium dioxides (PuO_{2-x}) and on mixed uranium-plutonium dioxides ($(U_{1-y}Pu_y)O_{2-x}$) were discussed. Previously the non-stoichiometric phase regions of these oxides were described as grossly non-stoichiometric single phase regions extending over considerable composition ranges. Contrary to this description the thermodynamic data obtained in this work clearly indicates that the non-stoichiometric phase region for these oxides can be divided into a number of "subphase regions", some of which are still non-stoichiometric with a characteristic type of defect cluster whereas others consist of a whole series of ordered intermediate phases. For the CeO_{2-x} system the structure of some of these phases has been determined in the present work by electron diffraction and high temperature X-ray measurements and the equipments used as well as the results obtained in these studies will also briefly be discussed.

Acoustic Emission and Carbide Cracking in Steel.

W.E. Swindlehurst, presented at the Meeting of the European Working Group on Acoustic Emission, Risø, September 1976. (Transcript available, 30 pp.)

Acoustic emission and carbide cracking in a low carbon ferrite/pearlite steel was followed as a function of strain in smooth tensile test pieces. Results indicate that carbide cracking is not a source of detectable emission in Risø tests.

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