



## Metallurgy Department. Progress report for the period 1 January to 31 December 1977

Risø National Laboratory, Roskilde

*Publication date:*  
1978

*Document Version*  
Publisher's PDF, also known as Version of record

[Link back to DTU Orbit](#)

*Citation (APA):*  
Risø National Laboratory, R. (1978). *Metallurgy Department. Progress report for the period 1 January to 31 December 1977*. Risø National Laboratory. Denmark. Forskningscenter Risoe. Risoe-R No. 377

---

### General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Risø National Laboratory

---

# Metallurgy Department Progress Report

for the period 1 January to 31 December 1977

DK7800048

March 1978

*Sales distributors:* Jul. Gjellerup, Sølvgade 87, DK-1307 Copenhagen K, Denmark

*Available on exchange from:* Risø Library, Risø National Laboratory, P.O. Box 49, DK-4000 Roskilde, Denmark

**INIS Descriptors**

**FUEL ELEMENTS**

**METALLURGY**

**NONDESTRUCTIVE TESTING**

**RESEARCH PROGRAMS**

**RISOE NATIONAL LABORATORY**

**UDC 669**

**March 1978**

**Risø Report No. 377**

**Risø National Laboratory**

**Metallurgy Department**

**Progress Report**

**for the period 1 January to 31 December 1977**

## **ABSTRACT**

The activities of the Metallurgy Department at Risø during 1977 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 1977 is included.

**ISBN 87-550-0517-9**

**ISSN 0418-6443**

---

## CONTENTS

	Page
Introduction .....	5
General Materials Research .....	8
Technology and Materials Development .....	14
Fuel Elements .....	17
Non-Destructive Testing .....	20
Participation in International Collaboration .....	23
Participation in the HI-77 Exhibition .....	24
Education and Training .....	25
Publications .....	26
Lectures and Conference Contributions .....	38
Staff .....	46

---



## INTRODUCTION

No political decision was taken in 1977 concerning the establishment of nuclear power stations in Denmark. Risø is therefore still at stand-by, and most of the nuclear programmes are being carried on in order to ensure continuously up-to-date knowledge in the field. In the Metallurgy department, nuclear work comprises projects such as the design and fabrication of fuel elements, in- and out-of-pile testing, post-irradiation examinations, and non-destructive testing of nuclear components. Of special interest are the newly designed LOWI (low-interaction) fuel pellets; irradiation of pins containing these pellets in the DR 3 reactor has given promising results. Many of the nuclear projects are carried out in collaboration with other parties in Denmark as well as abroad.

With respect to alternative energy technology, efforts were concentrated in three areas: fibre-reinforced composites, metal-hydrogen systems (e.g. for transport and storage of energy), and high-temperature ion conductors for batteries. A major project concerns the use of glass-fiber-reinforced plastic for windmill rotors. The department acts as a consultant to the Wind-power Programme of the Ministry of Commerce and Industry and to the electric utilities, who plan to put two 600 kW windmills into operation in 1979.



Work has been done under contract for industries and utilities in Denmark and abroad. Due to their proprietary nature, most of these activities are excluded from the present report. Among the major activities were fuel element development, where collaboration with the Elsinore Shipyard was continued, post-irradiation examinations of full-scale power-reactor fuel rods ( $Zr-UO_2$  and  $Zr-UO_2-PuO_2$ ) and isotope analysis. In the last two areas, much of the work took place as part of an EC programme on the recycling of plutonium in light-water reactors. A contract was entered into with the Danish utilities, ELSAM and Kraftimport, regarding the irradiation of  $Zr-UO_2$  fuel pins in DR 3 and fuel modelling.

Other work was done on high-temperature components for the chemical industry and acoustic emission for non-destructive testing purposes (the latter in collaboration with the Danish Welding Institute). Further projects were centered on the development of materials and processes; in particular problems relating to brazing were solved. A licence was granted to Krautkrämer-Branson in Germany for application of the Risø system for automatic measurement of the outside diameter of tubes, and a contract was signed with an American company for the sale in the USA and Japan of the fuel code WAPER 2 (developed in collaboration with the Elsinore Shipyard).

In an attempt to rationalize technological research in Denmark, a contract concerning brazing was signed with the Danish Welding Institute and the Corrosion Centre. According to this contract the Metallurgy Department performs research and development, whereas industrial application and training is the responsibility of the other parties. As part of the department's commercial activities, three senior staff members acted as part-time consultants to industrial enterprises.

To support the technological programmes of the department, much effort was devoted as usual to more fundamental problems, e.g., radiation damage in metals, strength-structural relations in single-phase and two-phase materials and structures in ceramics. A large part of this work is carried out in collaboration with universities and research laboratories in Denmark and abroad.

---

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

In order to demonstrate research techniques and results to the public, the department participated in an Industrial Exhibition (HI-77) held in Herning, Jutland. Equipment, films and posters were contributed to this event.

Educational activities were continued; students and post-graduates from Denmark and abroad studied in the department. One lic. techn. (Ph.D.) student passed his final examination during the year.

---

## GENERAL MATERIALS RESEARCH

Projects in this field are basic studies of the relationship between the structure and the mechanical properties of metals and other solid materials.

### Additive strength contributions

Tensile tests were carried out on copper with  $\text{Al}_2\text{O}_3$  particles to examine how the addition of strength contributions from particles and grain boundaries should be made. It was found that the grain boundary contribution was underestimated by simple linear or quadratic addition formulae. A weighted addition in which the grain boundary contribution was given more weight showed better agreement with the experimental results.

The specimens were fabricated by the earlier developed procedure for internal oxidation of Cu-Al in a  $\text{CO}/\text{CO}_2$  gas mixture. A method for control of the local Al content by

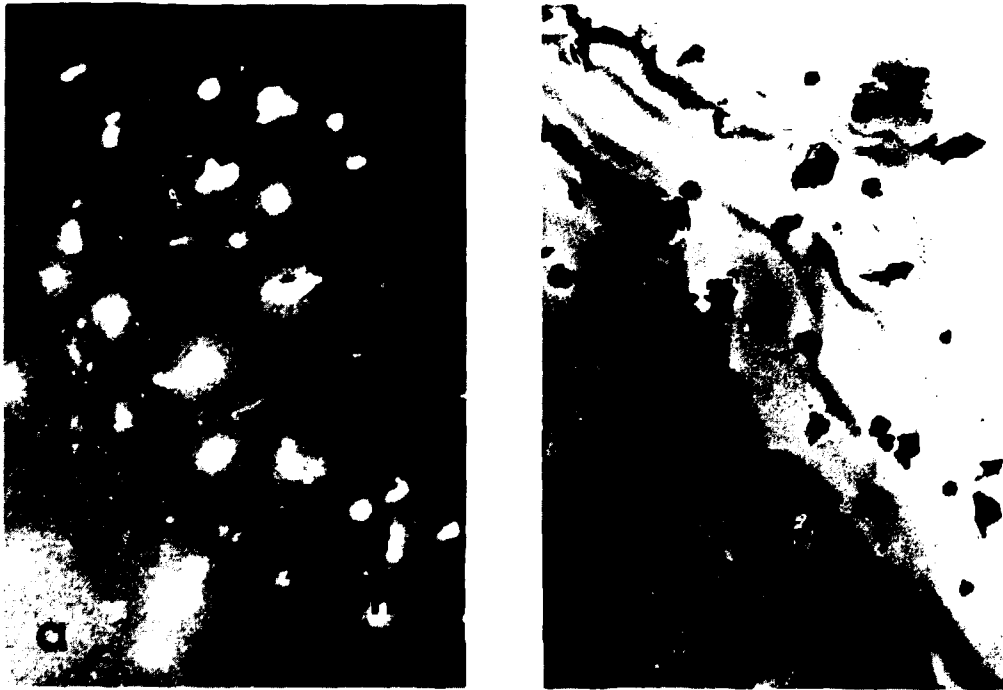


Fig. 1. Secondary emission (a) and conventional transmission (b) electron micrographs of  $\text{Al}_2\text{O}_3$  particles in Cu. Micrograph (a) is more suitable for automatic image analysis, but a comparison with (b) shows that a correction factor on the particle size must be introduced.

density measurements was developed. The particle size and volume fraction of  $\text{Al}_2\text{O}_3$  were determined from scanning electron micrographs (secondary emission mode) by means of automatic image analysis.

#### Deformation of composites

The mean field model (MFM) for the effective thermo-elastic and plastic properties of composites was compared with the established theory based upon Hashin and Shtrikman's variational principle. The model was found to reproduce existing variational results and to produce several results not yet obtained by variational methods. It can be concluded that the MFM is equivalent to the variational theory. The MFM is therefore also similar to the so-called concentric cylinder model for fibre composites: It is formulated on the basis of a rather artificial phase geometry (aligned ellipsoids); but it bounds the effective properties of composites with arbitrary microstructures. The MFM, initially formulated for two-phase systems, has now been extended to cover the general thermo-elastic and plastic behaviour of an N-phase composite and applied to the special case of the elasticity of random polycrystals. The model was found to improve the conventional Reuss and Voigt bounds. This was expected since, unlike the analyses of Reuss and Voigt, the MFM takes elastic grain interaction into account.

#### Creep in FCC metals

A model was suggested for the incubation period following a stress reduction during creep. Mobile dislocations are assumed to be produced by breakage of attractive junctions, and the incubation period is identified with the time needed to lower the breakage stress of the attractive junctions from the original stress to the new lower stress. The theory agrees well with experimental results found in the literature, and it explains the recovery rates found by the stress-drop technique.

A study of the dislocation structure in creep-deformed Cu-alloys was initiated.

### Fatigue phenomena in copper

A study was started of fatigue phenomena in single and polycrystals of pure Cu deformed in tension-compression at constant plastic strain amplitudes. An electronic device for control of plastic strain amplitudes and a grip system for the 25 kN MTS hydraulic testing machine were constructed.

### A finite element model of the deep-drawing process

The finite element computer program, developed to study the deep-drawing process and related metal-forming processes in axisymmetric situations, was improved. The program takes into account the geometrical and material nonlinearities arising as a result of the large displacements, finite strains, and plastic deformations involved in the metal-forming process. It also takes the friction forces between the tool and the blank into account. Results from the program were compared with experimental data from deep-drawing with a spherical-headed punch and good agreement was obtained.

### Radiation damage in stainless steel

(In collaboration with the Metallurgy Division, AERE Harwell, U.K.)

An austenitic stainless steel, resembling the commercial AISI type 316 steel, was made from high purity iron, chromium, nickel, and manganese. From the base material, several alloys containing Si and/or Ti were fabricated. Irradiation experiments were carried out in a high voltage electron microscope. Preliminary results on the alloy with 1.0% Si suggest that the swelling in the temperature range 450 to 600°C is drastically reduced as a result of Si addition.

### Radiation damage in pure copper

(In collaboration with the Metallurgy Division, AERE Harwell, U.K.)

High purity copper was irradiated in DR 3 at  $\sim 250^\circ\text{C}$  to fast neutron doses of  $10^{18}$  and  $5 \times 10^{18}$  n/cm<sup>2</sup>. Void density and

size were determined using a 100 kV electron microscope. Corresponding specimens were annealed, and positron life time and angular correlation measurements were made on these specimens after each annealing.

#### Radiation experiments on copper-nickel alloys

(In collaboration with the Metallurgy Division, AERE Harwell, U.K.)

The apparent activation energy was determined for the growth of interstitial dislocation loops in Cu and Cu-Ni during irradiation in the high voltage electron microscope. The resulting value for Cu and Cu-Ni with Ni contents up to 5% was  $0.35 \text{ eV} \pm 0.02 \text{ eV}$ ; for Cu-10% Ni, the result was  $0.43 \text{ eV} \pm 0.02 \text{ eV}$ .

For Cu, this closely corresponds to the value predicted by simple theory. Earlier experiments by other researchers have given somewhat lower values, but a theoretical analysis showed that this difference can be ascribed to a difference between our experiments and the earlier experiments in the thickness of the irradiated thin foils.

The fact that there is no significant increase in apparent activation energy for loop growth in Cu-2% Ni and Cu-5% Ni as compared to Cu indicates that the resistance to void swelling in Cu-Ni alloys containing 2% Ni and more is due to interstitial trapping (and not vacancy trapping). Very substantial interstitial trapping is needed to change the apparent activation energy for loop growth, whereas vacancy trapping would be immediately reflected in an increased apparent activation energy.

In an investigation of the effect of alloying elements on irradiation creep, Cu and Cu-10% Ni were reactor-irradiated (to a dose of approximately  $10^{20} \text{ n/cm}^2$ ) at  $185^\circ\text{C}$  without and with an applied stress ( $91 \text{ MN/m}^2$ ). After irradiation the structure was investigated by transmission electron microscopy. In the Cu specimens a dislocation network was under development, whereas the Cu-10% Ni specimens had a pure black-dot structure.

#### Radiation experiments on cold-worked pure copper

(In collaboration with the Metallurgy Division, AERE Harwell, U.K.)

High purity copper was cold-rolled at room temperature to thickness reductions of 10, 25, 50, 70 and 90%. Thin foils of the

different materials were irradiated in the high voltage electron microscope at 250, 300, 350 and 450°C.

At irradiation temperatures up to 350°C, the void number density was found to increase with the degree of cold-work up to a certain degree of cold-work and then to decrease again with further cold-work. Results at lower degrees of cold-work tend to suggest that areas of high dislocation density contained correspondingly high void density. At 450°C, the initial increase in void number density with cold-work was not very pronounced; the decrease in void density with further cold-work was, on the other hand, quite marked.

At irradiation temperatures up to 350°C there was no indication that cold-work produced any net reduction in void swelling. At 450°C, however, the swelling was substantially reduced for the high degrees of cold-work.

#### Blistering in stainless steel

(In collaboration with the Metallurgy Division, AERE Harwell, U.K.)

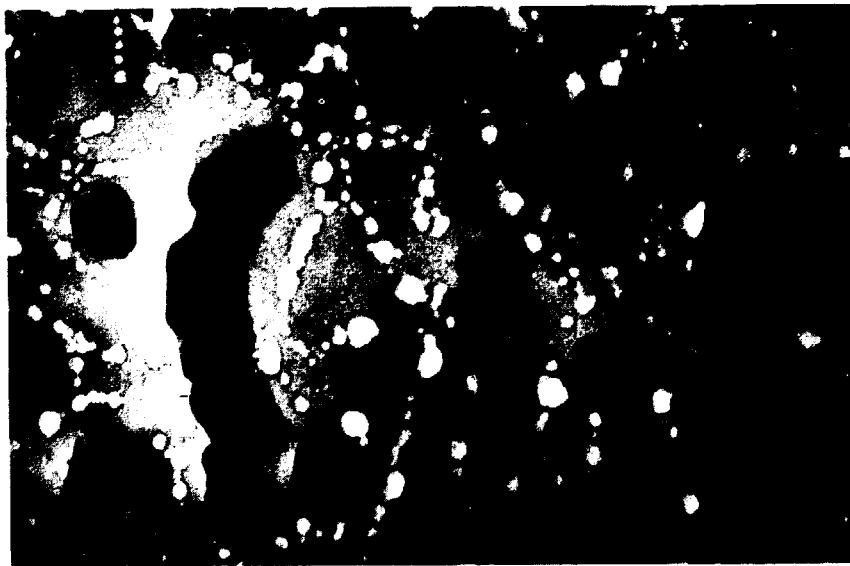


Fig. 2. Helium bubbles in 316 type stainless steel injected with  $5 \cdot 10^{17}$  He<sup>+</sup> (100 keV) at 500°C.

Specimens of stainless steels (type 316 and type 20 Cr-20 Ni, with and without dispersed particles) were bombarded with 100 keV He<sup>+</sup> ions at temperatures in the range 500 to 700°C. Two helium doses were examined;  $5 \times 10^{17}$  ions/cm<sup>2</sup> and  $10^{18}$  ions/cm<sup>2</sup>.

The surface and subsurface structures were examined by means of scanning electron microscopy and transmission electron microscopy, respectively.

Unexpectedly, no flaking was observed on any of the specimens. Small helium bubbles were commonly observed to be fairly randomly distributed in the matrix. It was also observed that at 600°C grain boundaries and particle-matrix interfaces were denuded of helium bubbles. In specimens bombarded at 500 and 600°C, large helium bubbles were observed at particle-matrix interfaces of large particles (complex carbide/oxide).

#### Recrystallization in dispersion-hardened metals

Experiments (in collaboration with the Department of Metallurgy and Materials Science at the University of Cambridge) on the nucleation and growth of recrystallization nuclei in Al-Al<sub>2</sub>O<sub>3</sub> were performed with special consideration of the nucleation at grain boundaries. Dynamic studies of the nucleation in Al containing FeAl<sub>3</sub> particles were made using the high voltage electron microscope at AERE, Harwell, UK. As a supplement to the recrystallization experiments, studies were made of grain growth after recrystallization in Al/Al<sub>2</sub>O<sub>3</sub> with different oxide contents.

#### Kinetic studies of oxidation and reduction of non-stoichiometric cerium oxides

Kinetic studies of CeO<sub>2-x</sub> were performed by isothermal and quasi-isothermal thermogravimetry in atmospheres with controlled oxygen pressures. In the quasi-isothermal technique, the programmed heating of the furnace is automatically stopped when the rate of the reaction (indicated by the derivative weight signal) exceeds a preset limit. The weight change curves suggest that both oxidation and reduction of CeO<sub>2-x</sub> are best described by the rate equation for a diffusion-controlled reaction. The experimental data further suggest that the CeO<sub>2-x</sub> phase region in the phase diagram consists of several subphases, characterized by their activation energy for the diffusion-controlled reactions.



## 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 methods were established for plastics reinforced with glass fibres and with carbon fibres. The methods are a simple leaky mould technique, and a filament winding technique for cylindrical specimens of up to two meters in length. A routine mechanical testing of fibre reinforced plastics was established for tensile and bending properties.



Fig. 3. Fracture region of an epoxy material reinforced with 60 vol% of long carbon fibres with approximately 10  $\mu\text{m}$  diameter. The matrix debris adhering to the fibres indicate a relatively good bonding between fibres and matrix.

Chemical plating with nickel of carbon fibre reinforced plastics was investigated. The preliminary results are promising.

The department acts as consultants to the electricity generating companies in connection with the building of power-generating wind turbines with financial support from the Ministry of Commerce and Industry. The consultancy concerns the selection of materials for the wing-blades, where glass-fibre-reinforced plastics are considered. The mechanical properties of laminates are calculated, and test samples of the wing design are investigated with respect to mechanical properties.

#### Brazing and soldering of aluminium

During contract work, industrial applications were developed from previous work on dip-brazing, vacuum brazing, and ultrasonic soldering of aluminium.

#### Metal-ceramic brazing

A study of metal-ceramic brazing was initiated with a literature survey and preliminary experiments on brazing of Fe-Ni alloys and high purity  $Al_2O_3$ . The aim of the experiments is to produce brazed joints that can withstand the combined influence of high vacuum and temperature cycling.

#### Joining method for fuel pins

The capacitor-discharge welding method was applied to the third closure of pressurized PWR fuel pins. A number of test welds were subjected to corrosion testing and metallographic examination. The results of the testing showed that the process is reliable and well suited for this purpose. A special miniature welding chamber was designed and constructed in order to make welding under pressure possible.

### DC-pulse welding of titanium sheet

A small number of test welds was made on titanium to investigate the influence of current-pulsing on the grain size and the ductility of the welds. No significant influence was found.

### Metal-hydrogen systems

Research was initiated on Mg-based alloys as media for hydrogen storage. Investigations of the main parameters such as temperature, pressure, surface area and surface contamination are in progress. Preliminary results indicate that temperature and pressure should exceed 350°C and 5 atm respectively, if a useful hydriding is to be obtained. There were indications that the addition of non-hydriding ductile metal addition (powder blending) was important in the low pressure - low temperature region.

## FUEL ELEMENTS

The Danish fuel elements in the Kahl and Halden reactors continue to perform well and to 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. Special tests such as power ramp tests are also being made.

Additional information on fuel performance becomes 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 the NRC (USA), and the EEC sponsored activities (Brussels) relating 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 9,800 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 26,200 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):

<u>IFA no.</u>	<u>148</u>	<u>161</u>	<u>165</u>	<u>201</u>	<u>202</u>
MWD/t $UO_2$	36,100	41,000	36,400	28,000	23,300

The maximum local burn-up of 51,300 MWD/t  $UO_2$  was achieved with IFA 161.

UO<sub>2</sub>-Zr irradiations at Risø

In the test fuel irradiation programme at DR 3, standard fuel pins have reached maximum burn-up levels of 46,800 and 35,600 MWD/t UO<sub>2</sub> for BWR and PWR fuel, respectively.

In the series of power ramp experiments, two more vipac pins and two pellet pins were tested at Risø, after completion of pre-ramp irradiation to 20,600 MWD/t UO<sub>2</sub> in an assembly in the Halden reactor. In addition, two PWR pins were ramp tested at 17,000 MWD/t UO<sub>2</sub>. Metallographic examination of previously ramp tested pins showed that stress-corrosion type cladding cracks can be very short axially, sometimes only one or a few wall thicknesses in length, as illustrated by fig. 4.

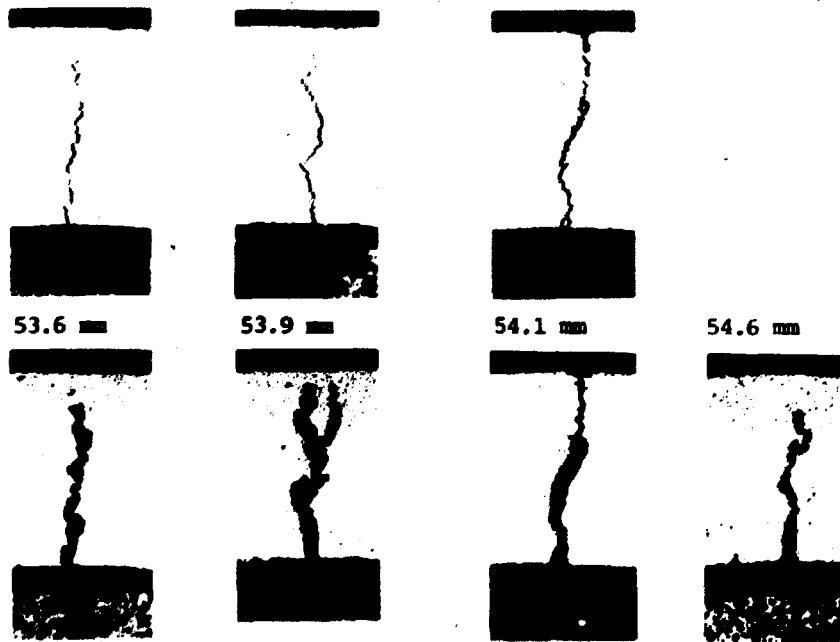


Fig. 4. Transverse sections at four axial locations in a fuel pin that failed in a ramp test. The pellet-pellet interface was between 54.1 and 54.6 mm. The cladding wall thickness was 0.6 mm. Upper pictures: as-polished, lower pictures: after hydride etching.

Evaluation of the special low-interaction (LOWI)  $UO_2$  pellet design was continued. One LOWI and one standard pin were irradiated to 10,600 MWD/t  $UO_2$  and then unloaded for non-destructive characterization prior to ramp testing early in 1978. Two other pins, again one LOWI and one standard pin, equipped with bellows for monitoring the internal gas pressure, were loaded into DR 3.

#### Computer modelling of fuel pin performance

The Danish fuel performance code WAFER was further developed. The present version, WAFER-2, includes features of the fast HOTCAKE codes (see the annual report for 1976) and a fuel restructuring model.

The code has been used to analyze a number of cases from EPRI's code evaluation project and the "Interramp" project. In both contexts, the WAFER results compared favourably with the experimental data and the outcome of other performance code analyses.

Contractual arrangements were made with a US consulting company regarding commercial exploitation of the WAFER code in the USA and Japan.

---

## NON-DESTRUCTIVE TESTING

The projects in this field deal with the development and application of non-destructive techniques for various testing purposes.

### X-ray technique

The image quality of three different types of X-ray papers was thoroughly investigated and compared with the image quality of conventional X-ray films. On the basis of this work it was possible to establish characteristic curves and exposure charts for different materials. It is concluded that the X-ray paper technique can be used in a wide area of X-ray investigations.

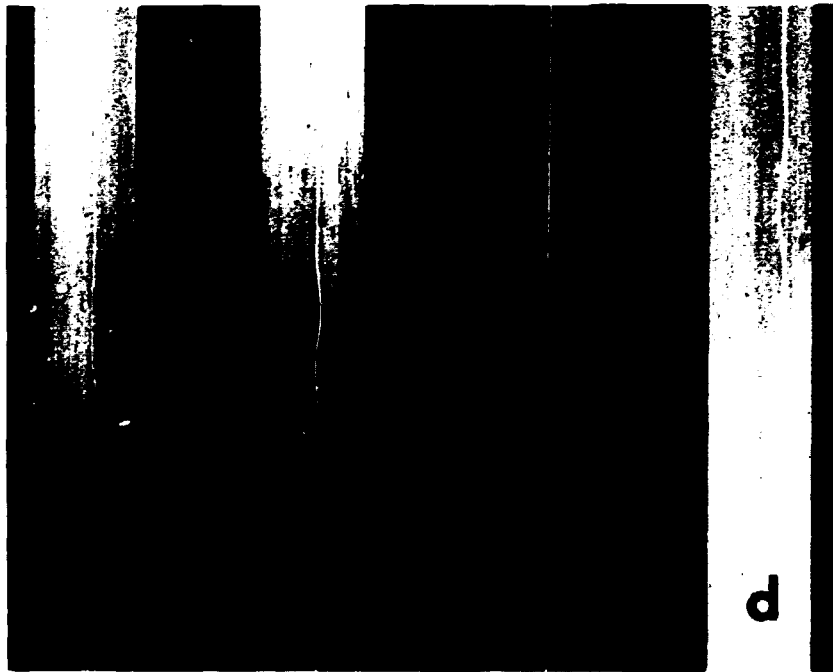


Fig. 5. X-ray radiographs of an epoxy material reinforced with 42 vol% of long carbon fibres. The samples contain natural defects, in particular elongated voids. Each sample contains a tungsten wire, the diameter of which is (a) 5  $\mu\text{m}$ , (b) 10  $\mu\text{m}$ , (c) 20  $\mu\text{m}$ , (d) 30  $\mu\text{m}$ .

The non-destructive examination of carbon-fibre-reinforced plastic by soft X-ray radiography was studied and the experimental conditions established. The smallest voids that can be recorded are about 100  $\mu\text{m}$  in size, while the smallest metallic inclusions (tungsten wire) that can be identified are 5  $\mu\text{m}$  in size.

The X-ray facility was rearranged and supplemented with an extra dark-room for the development of X-ray film and paper.

#### Neutron radiography

A new imaging material, nitrocellulose film, is being examined. A series of experiments with exposure time, development time and temperature used as variables was carried out. The results were compared with the results from the traditional direct or transfer techniques with X-ray films.

To improve the quality of images made by the new technique, the pictures are magnified on a Kodalith Ortho film and copied on Kodagraph Transfer paper. It is expected that a better resolution can be obtained for the neutron radiographs through the use of the new film material and the new technique.

#### Ultrasonic inspection

The experimental work on the tube inspection system was concentrated on the establishment of a better regulation of the water temperature. Work was started to establish an automatic scanning system for measurement of sound propagation from ultrasonic transducers to improve the characterization of these transducers. An investigation is in progress of the applicability of ultrasound as a tool for determining the structure and dimensions of composite materials.

---



### Acoustic emission

An investigation of the acoustic emission from two common structural steels is being made in collaboration with the Danish Welding Institute. Both base metal and welded test pieces are being investigated. Specimen temperature, loading rate, rolling direction, weld type, and post-weld heat-treatment are used as test parameters. The investigations have hitherto been conducted in the laboratory but it is planned to include experiments on pressure vessels.

The applicability of acoustic emission in evaluating the in-reactor performance of nuclear fuel pins is under study. Preliminary investigations of the problems associated with, for example, transducer mounting and mechanical/electrical noise isolation were completed. The actual acoustic response of fuel pins under power ramping and constant reactor power is being studied. Special emission analysis equipment was designed and will be built during 1978 in co-operation with the Electronics Department. Microstructural analysis of emission sources in a metal-matrix composite (Cu/W) is being made with a view to understanding emission mechanisms in composite systems of practical use.

### NDT in Hot Cells

A method for determining burn-up in irradiated fuel pins is under development. The axial distribution of relative burn-up is determined by gamma spectrometry. A calibration of the relative measure is obtained by making (destructive) isotope analysis on the fuel pin for a number of cross sections. A comparison of the results of this technique with results from the calorimetric measurements in the reactor showed good agreement.

The planning of a new combined fixture for complete NDT inspection of fuel pins in the hot cells is nearly finished. The new fixture will provide a comprehensive set of components that is well suited for remote operation and requires very little maintenance.

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

The department was represented on the following committees:

The Information Exchange Group under the European Space Agency on Carbon Fibre Reinforced Plastics,

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 HI-77 EXHIBITION**

Risø participated with a stand of 160 m<sup>2</sup> in the Industrial Exhibition which took place in Herning from 6-10 September 1977. The Metallurgy Department contributed posters, slides and working equipment to the stand.

Various techniques were illustrated; for instance ultrasonic soldering and vacuum brazing, both of which are more compatible with the environment than conventional techniques. The department's involvement in alternative energy research was demonstrated by posters illustrating fibre materials technology and hydrogen storage in metals.

The production of nuclear fuel elements for power reactors and test reactors was illustrated. Here use was made of films. One dealt with the production of uranium from Greenland ore, another followed the fabrication of the fuel elements that have been produced in collaboration with the Elsinore Shipyard, Ltd. for the Kahl reactor in Germany.

## 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 materials 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.

One scholarship holder from India worked in the Department on projects in the field of post-irradiation examination.

### Courses

Three courses were given in "Differential Thermal Analysis" with participants from Denmark, Norway and Sweden.

### 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:

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 Rods (in collaboration with the Reactor Department),
K.V. Rasmussen:	Fatigue Phenomena in Copper.

### Degrees conferred

The Technical University of Denmark conferred the degree of lic.techn. (Ph.D.) on C.P. Debel.

## PUBLICATIONS

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

Risø Report No. 354 (1977) 45 pp.

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.

Retningslinier for udarbejdelse af en virksomheds kvalitets-håndbog. (Guidelines to a Quality Control Manual).

B. Larsen, L. Bjerregaard Nielsen, S. Markmann, P. Buch Jensen, G. Nielsen, C.C. Agerup, and P. Drachmann, Studiekredsrapport nr. 7 (Dansk Forening for Kvalitetsstyring, 1977). 47 pp.

A Quality Control Manual is an authoritative collection of written procedures setting out how to perform the various tasks through which the company makes its products fit for use. These manuals are organized into modular sections, each dealing with some aspect of the quality function.

Creep Properties of Discontinuous Fibre Composites with Partly Creeping Fibres.

J.B. Bilde-Sørensen and H. Lilholt, Risø-M-1936 (1977) 19 pp.

In a previous report (RISØ-M-1810) we analyzed the creep properties of discontinuous fibre composites with non-creeping fibres. In the present report this analysis is extended to include the case of discontinuous composites with partly creeping fibres. It is shown, that the creep properties of the composite at a given strain rate,  $\dot{\epsilon}_c$ , depend on the creep properties of the matrix at a strain rate higher than  $\dot{\epsilon}_c$ , and on the creep properties of the fibres at  $\dot{\epsilon}_c$ . The composite creep law is presented in a form which permits a graphical determination of the composite creep curve. This can be constructed on the basis of the matrix and the fibre creep curves by vector operations in a  $\log \dot{\epsilon}$  vs.  $\log \sigma$  diagram. The matrix contribution to the creep strength can be evaluated by a simple method.

Dissociated Structure of Dislocation Loops with Burgers Vector  $a\langle 100 \rangle$  in Electron-Irradiated Cu-Ni.

J.B. Bilde-Sørensen, T. Leffers, and P. Barlow, Phil. Mag. 36 (1977) 585-595.

The rectangular dislocation loops with total Burgers vector  $a\langle 100 \rangle$  which are formed in Cu-Ni alloys during 1 MeV electron irradiation at elevated temperatures have been examined by weak-beam electron microscopy. The loop edges were found to take up a Hirth-lock configuration, dissociating into two Shockley partials and one  $a/3\langle 100 \rangle$  stair-rod dislocation. This explains the topography and the practical stability of the loops. Computer-simulated images of loops with the proposed structure agree with the experimental images. The behaviour of the rectangular loops seems to exclude the possibility that the Hirth lock can be an efficient barrier to dislocation motion.

Styrkemekanismer i Cu-legeringer. (Strengthening Mechanisms in Cu-Alloys).

P. Brøndsted, Risø-M-1958 (1977). Thesis. 99 pp.

A review is given of the relevant formulae for strengthening theories for Cu-alloys relevant to this project. A summary of results from earlier experiments is presented.

A method of fabricating homogeneous single crystals is analyzed and the homogeneity of Cu-Al single crystals is controlled by means of density measurements.

A new method for internal oxidation is described. The oxidation takes place in a CO/CO<sub>2</sub>-atmosphere and the oxygen pressure is controlled with a solid electrolyte oxygen cell. The relationship between the Al<sub>2</sub>O<sub>3</sub>-particle size and distribution and the oxidation parameters are determined experimentally.

Different mechanical testing methods are described and results from uniaxial tensile tests on Cu-Al<sub>2</sub>O<sub>3</sub>-alloys are used for verification of strengthening theories. Finally the addition methods of the strength contributions are investigated.

Welding and Brazing in Nuclear Engineering.

J. Christensen, Svejsning 4 No.3(1977) 75-77.

The Danish technological work within the nuclear field is mainly carried out in relation to the installations at the Risø National Laboratory, although some minor orders have been delivered to foreign nuclear power stations.

The main lines of this work, from where the following examples of welding and brazing jobs have been taken, are:

- development of tubular fuel elements for nuclear power stations,
- manufacturing of plate type nuclear fuel elements for materials testing reactors, and
- fabrication of equipment for research reactor installations, e.g. irradiation facilities.

**Dynamisk brudmekanik. (Dynamic Fracture Toughness).**

C.P. Debel, Risø-M-1897 (1977). Thesis. 200 pp.

A review of the concepts of quasi-static and dynamic fracture mechanics is given, and the method of obtaining the dynamic toughness  $K_{ID}$  of a material as proposed by Battelle, Columbus Laboratories, is described in detail.

Experimentally the quasi-static and dynamic toughness of a plain carbon steel was examined using the concepts of fracture mechanics. Wedge-loaded DCB-specimens were used in dynamic tests, and the "equivalent-energy" method was used in evaluating the quasi-static toughness. A new kind of surface-deposited grid has been developed and used together with a new type of electronic circuit in order to measure the velocity of rapidly propagating cracks.

Specimens were tested at temperatures between  $-115^{\circ}\text{C}$  and  $+22^{\circ}\text{C}$ . The lower temperatures are obtained by cooling the specimen in liquid nitrogen and allowing the specimen to heat up until test temperature is reached. The distribution of temperature in the specimen that results using this procedure has been examined and is shown to be small if insulation of the specimen is used shortly before the test.

Rapid crack propagation produces a substantial shift in the toughness transition region to higher temperatures, and the dynamic toughness decreases with increasing crack velocity at a given temperature. Large crack-driving forces result in heavily branched cracks and low overall velocity of propagation, whereas small crack-driving forces produce high velocities and fracture surfaces of smooth appearance.

**Experimental Evaluation of Brittle Crack Propagation Velocity - an Improved Technique.**

C.P. Debel, Risø-M-1961 (1977) 17 pp.

A short review of experimental methods currently used in evaluating the velocity of fast crack extension is given. The technique of applying a surface deposited gridgauge has been innovated. This new technique involves a grid produced by a photo-chemical method and an electronic registration circuit based on integrated transistor-transistor logic. This new method has successfully been applied to experimental studies of brittle crack extension in steel at temperatures between  $-115^{\circ}\text{C}$  and  $+22^{\circ}\text{C}$ .

**Brudmekanik og Procesværktøjer. (Fracture Mechanics applied to Tools).**

C.P. Debel, In: Varmebehandling og Værktøjsfremstilling, Dansk Metallurgisk Selskabs Vintermøde, Bjerringbro, 3-5 January 1977. Edited by V.F. Buchwald. (Dansk Metallurgisk Selskab, København, 1977) 285-300.

The concepts of linear-elastic fracture mechanics are discussed, and examples of calculation are given with regard to tools used in the mechanical processing of metals. Fatigue loading is also considered.

### Industrial Radiography on Radiographic Paper.

J.C. Domanus, Risø Report No. 371 (1977) 183 pp.

A review is given of the rather scarce literature on the subject. An investigation was performed to compare the quality of radiographic paper with that of X-ray film. The equipment used throughout the investigation is described, and characteristic curves for Agfa-Gevaert and Kodak papers exposed with different intensifying screens in the low and intermediate voltage range are reproduced. The relative speed, contrast and exposure latitude were computed from these curves. The quality of the radiographic image was checked on U/Al blocks and plates, Al and Fe blocks, and fiber-reinforced composites. Exposure charts for Al and Fe were produced for various paper and screen combinations. A check was made on the sharpness of the radiographic image, as well as on the influence of processing on speed and contrast. Examples are given of the practical applications of the paper for radiography of castings, weldings, solderings, assemblies and some other unusual applications.

### Activities in Neutron Radiography at Risø, Denmark.

J.C. Domanus, Neutron Radiography Newsletter 15 (1977) 3-6.  
(Also as Risø-M-1955 (1977) 6 pp.).

The DR1 reactor at Risø is used as a neutron source for neutron radiography. In the doublebeam neutron radiography facility a neutron flux of an intensity of  $1.4$  and  $1.8 \times 10^6 \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$  reaches the object to be radiographed.

The transport and exposure container used for neutron radiography of irradiated nuclear fuel rods is described, and the exposure technique and procedure are reviewed. The mode by which single neutron radiographs are assembled and assessed is described.

### Radiographic Control of Materials Testing Reactor Fuel on X-ray Paper.

J.C. Domanus, Nucl. Technol. 36 (1977) 187-192.

The introduction of x-ray paper for the radiographic control of Materials Testing Reactor fuel was preceded by an investigation of the sensitometric properties of Kodak and Agfa-Gevaert paper at 45 and 150 kV.

Radiographic quality of aluminum, iron, and uranium/aluminum block and uranium/aluminum plate radiographs showed that the x-ray paper has adequate quality (image quality indicator sensitivity better than 2%) and therefore can be used instead of x-ray film, which is more expensive and requires longer exposure and processing time.



**Dislocation and Grain Boundaries - A Molecular Dynamics Investigation.**

P.O. Esbjørn, Risø-M-1944 (1977). Thesis. Vol. I + II, 226 + 148 pp.

Defect structures in a two-dimensional Lennard-Jones crystal are simulated using the molecular dynamics method. Dislocation parameters of a stationary dislocation are derived comparing results for the discrete model with those derived from elastic continuum theory. For a dislocation moving under applied shear the critical stress for dislocation motion, the Peierls stress, and the height of the corresponding potential barrier are determined. The structure and energy of grain boundaries are calculated as a function of misorientation. The grain boundaries are generated by solidifying a liquid between two mutually misoriented slabs of solid crystals and also by directly sticking together two solid half crystals. The migration of a high-angle grain boundary acting as a sink for moving interstitials is studied introducing interstitials in the central part of the central grain of a tricrystal. Mechanisms for transmission of plastic deformation from one grain to another in a polycrystal are investigated by simulations of interactions between a high-angle grain boundary and different combinations of dislocations moving under applied shear.

**A New Ultrasonic Inspection System for Non-Destructive Examination of Precision Tubes. Part 1. A Description of the System.**

H.E. Gundtoft, C.C. Agerup, and T. Nielsen, NDT Int. 10 (1977) 171-176.

The Risø/HV Tube Inspection System is designed for fast automatic inspection of the dimensions of and defects in precision tubes. The system is based upon a rotating water chamber with eight ultrasonic transducers. Tube handling and evaluation of the results can be performed in various ways using different combinations of the modules in the system. This paper describes evaluation and development work on this system that has been carried out, based on practical experience with tube inspection. Also discussed is the influence of various modifications to the system on stability and accuracy during actual tube inspection, which was studied by repeated inspection of the same 17 tubes.

**A New Ultrasonic Inspection System for Non-Destructive Examination of Precision Tubes. Part 2. Computer Control.**

H.E. Gundtoft and N. Nielsen, NDT Int. 10 (1977) 177-180.

In the automatic inspection system used by the Research Establishment at Risø - described in Part 1 - data (more than half a million per tube) from the ultrasonic dimension measurement and defect inspection are fed into a computer that simultaneously calculates and evaluates the results. This paper describes the development of the computer program used and discusses the advantages of computer control over normal analogue recording techniques. The overall benefits gained by using a computer controlled ndt system are outlined.

**Erfahrungen mit einem neuen Ultraschallkontrollsystem für die zerstörungsfreie Prüfung von Präzisionsrohren.**

**H.E. Gundtoft, T. Nielsen, and C.C. Agerup, Materialprüfung 19 (1977) 385-388.**

The complete Tube Inspection System for detection of defects and determination of dimensions includes a minicomputer, which controls an automatic sorting equipment, so that a fully automatic inspection can be made. This paper describes the experiences and the improvements which have resulted in a system with sufficient stability and accuracy to perform inspection to the most stringent specification values for the dimension- and defect control of precision tubes. In the standard examination of canning tubes the total examination for defects and dimensions can be carried out with a speed of 6 meter tube per minute.

**The Effect of Grain Size and Strain on the Tensile Flow Stress of Aluminium at Room Temperature.**

**N. Hansen, Acta Met. 25 (1977) 863-869.**

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.

**Indledende komponent- og konceptstudier for vindkraftforsøgsanlæg, afsnit 5.2.3.: Materialer, konstruktion og fremstilling. (Introductory Studies of Components and Concepts for Research Wind Mills, part 5.2.3.: Materials, Construction, and Manufacture).**

**B.S. Johansen, Aa. Lystrup, and H. Lilholt, In: Indledende komponent- og konceptstudier for vindkraftforsøgsanlæg. (Handelsministeriets og Elværkernes Vindkraftprogram, Lyngby, 1977) 64-86.**

A brief description is given of materials, which may be used in the construction of wing-blades for large wind-turbines. The materials reviewed are steel, aluminium, wood, glass-fibre reinforced plastics, and carbon-fibre reinforced plastics. Design concepts for the wing-blades are discussed, and available fabrication methods are discussed with particular reference to their cost.

## A New Version of the LWR Fuel Performance Model WAFER.

N. Kjør Pedersen, In: Transactions of the 4th International Conference on Structural Mechanics in Reactor Technology, San Francisco, 15-19 August 1977. Vol. D. Edited by T.A. Jaeger and B.A. Boley (Commission of the European Communities, Luxembourg, 1977). Paper D1/3, 11 pp.

This contribution discusses the latest version, WAFER-2, of the Danish LWR fuel performance code WAFER. The original version, WAFER-1, has previously been published. Modification in the new version are:

- (i) Restrictions on fuel crack penetration have been completely removed. This implies more accurate stress analysis, hence more realistic creep calculations.
- (ii) Several transversal fuel cracks at arbitrary axial locations (as opposed to one at the mid-pellet position in WAFER-1) are now permitted. This further improves stress analysis and renders the pellet model duly sensitive to interaction forces from the cladding.
- (iii) The total radial crack volume is now evenly distributed circumferentially. This insures better accuracy in the average stress/strain values, at the cost of insignificant local detail.
- (iv) Elastic moduli and other materials properties are now functions of temperature and other operational parameters.
- (v) The fundamental mode of calculation has been changed: WAFER-1 calculates time step increments of all system variables, to be added to base values from the preceding time point. WAFER-2 does an absolute base value calculation at each time point for all non-integrator variables. This change saves storage and reduces error accumulation.
- (vi) A series of new capabilities has been added to the code: Densification, gaseous swelling and fission gas release.
- (vii) The treatment of primary creep and yielding of the cladding has been improved.

## Ramp Testing of $\text{UO}_2$ -Zr Fuel Pins up to 29000 MWD/te $\text{UO}_2$ . P. Knudsen, Trans. Am. Nucl. Soc. 27 (1977) 244-245.

Three almost identical test fuel pins were irradiated in water-cooled facilities at 70 at. First, all pins were mounted together and irradiated to a burnup of 26,800 MWD/te  $\text{UO}_2$  over a period of about 3 years. The heat load was generally decreasing in the range 560 to 280 W/cm, the latest level being 360 W/cm.

Following non-destructive characterization, the power of one of the pins (M20-1B) was increased to 470 W/cm at a rate of 27 W/cm·min. The new level was maintained for 550 hrs without failure indication.

The other two pins (M2-2B and T9-3B) were then further irradiated to 29,000 MWD/te  $\text{UO}_2$  in the range 250-270 W/cm, the latest level being 260 W/cm. The pins were re-characterized and the power increased to 470 W/cm at the same rate as above. The pins were unloaded after a 670 hrs hold-time without failure indication.

### Characterization of PWR Ramp Tests.

P. Knudsen, C. Bagger, and M. Fishler, In: ANS Topical Meeting on Water Reactor Fuel Performance, Proceedings of a Meeting held at St. Charles, Ill., 9-11 May 1977. (American Nuclear Society, 1977) 243-252.

Three identical PWR type test fuel pins were irradiated to 11,200 MWD/te  $UO_2$  at heat loads decreasing from 510 to 340 W/cm (avg. test levels). Following non-destructive characterization, the power level was increased to 540 W/cm, at a rate of 20 W/cm-min. This caused two of the pins to fail, whereas the third survived an additional increase of 50 W/cm. The detailed post-test examination revealed cladding failures that were similar in appearance to those usually attributed to a stress-corrosion mechanism.

### Dislocation Climb Sources Activated by 1 MeV Electron Irradiation of Copper-Nickel Alloys.

P. Barlow and T. Leffers, Phil. Mag. 36 (1977) 565-583.

Climb sources emitting dislocation loops are observed in Cu-Ni alloys during irradiation with 1 MeV electrons in a high voltage electron microscope. High source densities are found in alloys containing 5, 10 and 20% Ni, but sources are also observed in alloys containing 1 and 2% Ni. The range of irradiation temperatures corresponding to the highest source densities is approximately 350<sup>o</sup>-500<sup>o</sup>C. The climb sources are not related to any pre-existing dislocations resolved in the microscope. The sources emit three types of loop: 'rectangular' loops with a <100> Burgers vector and {100} habit plane, normal prismatic loops with Burgers vector  $a/2\langle 110 \rangle$ , and Frank loops. There is no significant difference between the apparent activation energy for growth of the three types of loops.

The source points are suggested to be submicroscopic nickel precipitates - with reference to the existing evidence that, thermodynamically, there is not complete miscibility in the Cu-Ni system as implied by the published phase diagrams. It is furthermore suggested that these precipitates are platelets of Ni atoms on {100} planes, which would account for the formation of the rectangular loops. The binding energy between vacancies and Ni precipitates in Cu-10% Ni is estimated to be 0.3 eV.

### Rectangular $a\langle 100 \rangle$ Loops in Electron Irradiated Cu-Ni.

T. Leffers, J.B. Bilde-Sørensen, and P. Barlow, Scand. J. Met. 6 (1977) 29-30.

The abstract appeared in the previous progress report p. 41.

## Void Formation and Growth in Copper-Nickel Alloys During Irradiation in the High Voltage Electron Microscope.

T. Leffers, B.N. Singh, and P. Barlow, Risø-M-1937 (1977)

38 pp.

The formation and growth of voids during irradiation in a high voltage electron microscope were studied in copper and Cu-Ni alloys. For each composition the range of irradiation temperatures from 250°C to 550°C was covered. The development of the irradiation-induced dislocation structure was also studied. At irradiation temperatures up to 450°C the void swelling decreased rapidly with increasing Ni content and became practically zero for Cu-10% Ni. The decrease in swelling was produced mainly by decreased void growth (and not by decreased void number density). At 550°C the void swelling increased with increasing Ni content up to 5%, whereas for Cu-10% Ni the swelling became practically zero; again the changes in swelling with Ni content were mainly determined by changes in void growth.

The reduction in void swelling and growth due to alloying is ascribed to vacancy or interstitial trapping at submicroscopic Ni precipitates, i.e. to the precipitates acting as recombination centres. The increase in void swelling and growth with increasing Ni content, on the other hand, is ascribed to dislocation climb sources which emit loops and hence produce a fairly high dislocation density at a temperature where there are only few dislocations in pure copper or Cu-Ni with lower Ni content.

## Hardening in Two-Phase Materials - I. Strength Contributions in Fibre-Reinforced Copper-Tungsten.

H. Lilholt, Acta Met. 25 (1977) 571-585.

Cyclic tests (Bauschinger tests) were conducted at 77 K and at room temperature on the fibre-reinforced material of single crystal Cu with long W-fibres of diameter 20 µm and volume fractions up to 4%. These tests enabled two important contributions to the total strength of the unrelaxed material, the mean stress and the source-shortening stress, to be determined. The mean stress was measured independently, and was found to be a unique function of the numerical applied strain, both at 77 K and at room temperature. The theory for the mean stress describes fairly accurately the behaviour at 77 K for strains below 0.5%. The behaviour at room temperature for strains below 0.4% was not in quantitative accord with the theory, and it is suggested that a reversible relaxation takes place by one or several hindered dislocation processes. The crystallographic features were found to have only a minor influence on the mean stress. The source-shortening stress was estimated as the difference between the total stress and the mean stress; empirical equations showed a dependence on strain to the power  $\frac{1}{2}$  and on volume fraction plus a constant; these features are not included in the simple model which however predicts numerical values of the correct order of magnitude. The cyclic hardening rates were large compared to, e.g. pure Cu, and they were constant within the experimental limits. At larger strains, about 0.5% at 77 K and 0.4% at room temperature, irreversible relaxation of Cu-W was initiated by yield of the fibres. This strain for yield is lower than the strain for the unhindered relaxation in the dispersion-hardened material Cu-SiO<sub>2</sub>.

**Hardening in Two-Phase Materials - II. Plastic Strain and Mean Stress Hardening Rate.**

H. Lilholt, *Acta Met.* 25 (1977) 587-593.

The strain parameters which are relevant in a tensile experiment, are analysed and related to the geometry of deformation and to the mean stress of two-phase materials. The hardening rate of the mean stress with respect to plastic strain is found to be useful in comparison between experiments and theories, and it allows theories to be probed over a range of strains. Previous experiments on the fibre-reinforced material of copper-tungsten are analysed in relation to the geometry of deformation.

**Creep-Rupture Properties and Corrosion Behavior of  $2\frac{1}{4}$  Cr-1 Mo Steel and Hastelloy X Alloys in Simulated HTGR Environment - Interim Report.**

Aa. Lystrup, P.L. Rittenhouse, and J.R. DiStefano, ORNL/TM-6001 (1977) 42 pp.

Hastelloy X and  $2\frac{1}{4}$  Cr-1 Mo steel are being considered as structural alloys for components of a High-Temperature Gas-Cooled Reactor (HTGR) system. Among other mechanical properties, the creep behaviour of these materials in HTGR primary coolant helium must be established to form part of the design criteria. This report describes the simulated HTGR-helium environmental creep facilities, summarizes preliminary creep properties of  $2\frac{1}{4}$  Cr-1 Mo steel and Hastelloy X generated in HTGR helium and compares these with data obtained by testing in air. Some corrosion characteristics of the two materials are also discussed.

**Equivalence of Stress- and Energy Calculations of Mean Stress.**

O. Bøcker Pedersen and L.M. Brown, *Acta Met.* 25 (1977) 1303-1305.

Calculations of the mean stress in a plastically deformed matrix containing randomly distributed elastic inclusions are considered. The mean stress for an elastically homogeneous material is calculated on the basis of an energy consideration which completely accounts for elastic interactions. The result is shown to be identical to that obtained from a stress calculation. The possibility of including elastic interactions in the case of elastic inhomogeneity is discussed.

Atmosfærekontrol ved Varmebehandlinger ved Hjælp af Elektrokemiske Måleceller baseret på Faststof-Ionledere. (Control of Furnace Atmosphere with Electrochemical Cells based on Solid Ionic Conductors).

O. Toft Sørensen, In: Varmebehandling og Værktøjsfremstilling, Dansk Metallurgisk Selskabs Vintermøde, Bjerringbro, 3-5 January 1977. Edited by V.F. Buchwald. (Dansk Metallurgisk Selskab, København, 1977) 181-192.

The theoretical background for this technique, the principle of a cell developed at the Risø Metallurgy Department for furnace atmosphere control and some typical uses of this technique in metallurgical research are described. The technique can also be used for measurement of oxygen potentials in molten metals and some examples (from the literature) of this are discussed.

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

O. Toft Sørensen, In: Reactivity of Solids. Proceedings of the 8th International Symposium on the Reactivity of Solids, Göteborg, 14-19 June 1976. Edited by J. Wood, O. Lindqvist, C. Helgesson, and N.-G. Vannerberg. (Plenum, New York and London, 1977) 659-662.

The abstract appeared in the previous progress report p. 42.

Scandinavian and International Co-operation within Thermal Analysis.

O. Toft Sørensen, Kem.-Kemi 4 (1977) 385-386.

In order to promote communication between all scientists working with thermal analysis in the Nordic countries, the Nordic Society for Thermal Analysis (NSTA) has been formed. A short review of the history, organization and activities of this Society is given, as well as of its co-operation with the large international organization for thermal analysis, the International Confederation for Thermal Analysis (ICTA).

Måske får vi brintdrevne biler, der lades op fra husledningen.  
(Shall we see hydrogen-generated cars charged from household  
gas supply).

B. Vigeholm, ingeniøren 3 No. 50 (1977) 29-30.

A survey of the hydride energy concept is given. The metal-hydrogen reaction and general temperature-pressure dependencies is outlined. Energy-relevant applications are mentioned with some elaboration on mobile energy storage, utilization of low-temperature waste heat and hydride heat pumping.

#### Scanuk: A Collaborative Programme to Develop New Zirconium Cladding Alloys.

C. Tyzack, P. Hurst, G.F. Slattery, F.W. Trowse, A. Garlick, R. Sumerling, A. Stuttard, K. Videm, L. Lunde, M. Warren, E. Tolksdorf, P. Tarkpea, and J. Forsten, J. Nucl. Mat. 66 (1977) 163-186.

The primary aim of the programme was to develop alloys with better performance than Zircaloy-2 under BWR/PWR conditions and specifically with improved resistance to short-term high temperature transients. Secondly the alloys were to be capable of full-term reactor service over a wider temperature range than usual (up to 450°C). For the first objective a Zr-1 wt% Nb alloy was selected, and for the second, alloys were composed of small amounts of chromium and/or molybdenum added to a base composition of 0.5 or 1 wt% niobium in zirconium. This paper describes the test programme and results obtained on the physical metallurgy, mechanical properties and corrosion resistance of the alloys both before and after irradiation. Although the requirement for cladding to operate at elevated temperatures is no longer of prime importance the development work has demonstrated that with some further optimisation some of the alloys might present a viable alternative to Zircaloy-2 for in-reactor operation at  $\approx 300^{\circ}\text{C}$  in oxygenated coolants. Especially with regard to nodular oxidation resistance these alloys based on modest additions of niobium to zirconium tend to be better than Zircaloy-2 but their performance does not consistently approach that of Zr-2.5% Nb.



## LECTURES AND CONFERENCE CONTRIBUTIONS

Kursus i Legeringslære. (A Course in the Structure and Properties of Alloys).

C.C. Agerup, presented to Dansk Forening for Materialografi, Jydsk Teknologisk Institut, November 1977. (Not available).

Non-Destructive Testing of Five Fuel Rods Irradiated in Garigliano BWR up to 20,000 MWD/t.

C. Bagger, presented to the EEC Plutonium Recycling Group, Bruxelles, June 1977. (Not available).

Non-destructive examination of five LWR oxide fuel rods from the Garigliano power station has been performed at the Risø National Laboratory. Four of the rods contained fuel with initially 0.82% to 3.2% Pu in natural uranium oxide, the fifth rod to be regarded as a reference rod was enriched in uranium. The burn up level is estimated to 20 GWd/t  $\text{MeO}_2$ .

None of the extensive examinations indicate the presence of potential failures. It may be concluded on a preliminary basis that the performance of the rods up to a burn up level of 20 GWd/t  $\text{MeO}_2$  has been satisfactory.

Post-Irradiation Examination of IFA 226 - Results and Preliminary Evaluation.

C. Bagger, H. Carlsen, H. Hougaard, E. Larsen, and N. Larsen, presented at the Enlarged Halden Programme Group Meeting, Sanderstølen, March 1977. (Not available).

The instrumented, initially high plutonium enriched test fuel assembly IFA 226 was irradiated to a burn-up of 30 GWd/t  $\text{MeO}_2$  in the Halden Boiling Water Reactor under the USNRC's participation in the OECD Halden Reactor Project. The results of an extensive post irradiation examination carried out at Risø are presented and discussed on a preliminary basis.

## Influence of Surface Treatments on the Shear Strength of Brazed Joints in Inconel X-750.

J. Christensen, presented at the International Institute of Welding High-Temperature Colloquium, København, July 1977. (Proceedings to be published).

This paper describes an investigation in which it is found, that the shear strength of joints in Inconel X-750 nickel brazed in a getter atmosphere at 1025°C and a vacuum of only  $10^{-2}$  torr are equal to or better than the shear strength obtained on "untreated" specimens brazed at at least a 50°C higher brazing temperature and in a high vacuum of  $10^{-5}$  torr or better. This agrees well with earlier results where a perfect wetting was found at exactly the same conditions.

## Erfahrungen mit einem neuen Ultraschallkontrollsystem für die zerstörungsfreie Prüfung von Präzisionsrohren.

H.E. Gundtoft, T. Nielsen, and C.C. Agerup, presented at the Vortragsstagung 1977, Deutsche Gesellschaft für Zerstörungsfreie Prüfung, Bremen, May 1977. (Manuscript published in Materialprüfung).

## The LOWI Fuel Design, Test Results and Calculations.

A. Jensen, presented at the Enlarged Halden Programme Group Meeting, Sanderstølen, March 1977. (Not available).

Irradiation rigs containing fuel pins of the new LOWI fuel design are now operating in the DR 3 test reactor at Risø. One rig was unloaded after one reactor period, 22 days, and transferred to the Hot Cells for examination. Results are presented from this first irradiation experiment together with calculations performed with the fuel performance code "HOTCAKE, TR". Included are also calculations made on a reference basis of some reactor-physical properties and cladding transients in a LOCA.

## Analytical Verification and its Limitations.

N. Kjar-Pedersen, presented at a Seminar on Fuel Rod Mechanical Modeling, San Francisco, August 1977. (Transactions to be published).

A number of physical concepts used in fuel modelling are reviewed; their validity and interrelation and the possibility of experimental verification are discussed.

### Performance Evaluation of $UO_2$ -Zr Fuel In Power Ramp Tests.

P. Knudsen, C. Bagger and N. Kjær-Pedersen, presented at the International Conference on Nuclear Power and Its Fuel Cycle, Salzburg, May 1977. (Proceedings to be published).

A test fuel element containing both pellet and vipac  $UO_2$ -Zr fuel pins was irradiated in the HBWR at Halden for effectively 2½ years to an average burn-up of 21,000 MWD/te  $UO_2$  at gradually decreasing power levels. The subsequent non-destructive characterization revealed formation of transverse cracks in the vipac fuel columns. After the HBWR irradiation, five of the fuel pins were power ramp tested individually in the DR 3 Reactor at Risø.

The observed failures seemed to be marginal since little or no indication as to the locations of the clad penetrations could be derived from the non-destructive post-irradiation examinations.

The cases have been analyzed by means of the Danish fuel performance codes. The calculations, which are in general agreement with the observations, are discussed in the paper.

### Danish Developments Related to Water Reactor Fuel Performance and Technology.

P. Knudsen, presented at the IAEA Working Group on Water Reactor Fuel Performance and Technology, Wien, September 1977. (Transcript available, 7 pp.).

This note presents a resumé of the Danish  $UO_2$ -Zr irradiations (burnup levels are as of 1 July 1977) and performance code development. Reference is also made to a new pellet design LOWI, developed for minimum pellet-clad interaction, and examples are mentioned where special techniques developed during the above work have been of interest in other contexts.

### Danish Investigations Related to Fuel-Clad Interaction.

P. Knudsen, presented at the IAEA Specialist Meeting on Pellet-Cladding Interaction, Wien, June 1977. (Transcript available, 24 pp.).

Detailed metallographic observations from a BWR ramp test are presented and compared with previously published results from PWR ramp tests. It is concluded that pin failure most likely was caused by a stress-corrosion mechanism. Results with two vipac tests indicate that this fuel type may have a ramp performance superior to that of conventional pellet design. Initial testing of a new pellet design suggests that this can reduce pellet-clad interaction considerably.

**Fission Gas Release from Very High Burnup UO<sub>2</sub>-Zr Fuel Pins.**

**P. Knudsen and H. Carlsen, presented at the Enlarged Halden Programme Group Meeting, Sanderstølen, March 1977. (Not available).**

Two UO<sub>2</sub>-Zr test fuel pins were irradiated to burnups exceeding 40,000 MWD/te UO<sub>2</sub> at heat loads in the range 400-600 W/cm, with latest levels around 500 W/cm. After non-destructive characterization, the fission gases released to the internal pin voidage were extracted and analyzed.

The irradiation was simulated with a Danish fuel performance code which has options for several fission gas release models. The paper presents the experimental results and evaluates the predictive capability of the gas release models at this very high burnup level.

**Separation af nogle sjældne jordarter ved ionbytnings-HPLC. (Separation of some rare-earth ions by high-pressure liquid chromatography).**

**N. Larsen and W. Batsberg Pedersen, presented to the chemical engineering group, Dansk Ingeniørforening, November 1977. (Not available).**

Anion-exchange chromatography of the rare-earth (Sm, Nd, Pr, Ce) ions in methanol/nitric acid/water media is performed using high-pressure liquid chromatography. The separation method is especially designed for determination of <sup>148</sup>Nd in the nuclear fuel cycle in order to find precise burn-up values. The high-pressure liquid chromatography method presented here is very fast, gives high resolutions, and enables collection of selected fractions containing n moles of rare-earth by UV-monitoring at 280 nm of the eluate.

**The Taylor and the Sachs Model for the Plastic Deformation of Polycrystals seen in the Light of Dislocation Theory.**

**T. Leffers, presented to the Metal Physics Group of the Roland Eötvös Physical Society, Budapest, September 1977. (Not available).**

There is a general trend, mainly based on continuum-mechanistic considerations, to prefer the Taylor theory for the Sachs theory for the description of polycrystal plasticity. It is shown, on the basis of simple dislocation theory and computer-simulation of the interaction of dislocations with grain boundaries, that a modified Sachs theory is much more compatible with the deformation pattern of real polycrystals - from a theoretical as well as an experimental point of view.

### Transformation Theory for Composites.

O. Bøcker Pedersen, presented at the GAMM/DCAMM Congress 1977, Lyngby, June 1977. (Manuscript to be published in Zeitschrift für Angewandte Mathematik und Mechanik).

The dielectric problem is considered in its elastostatic form. Simple mean field analyses on the basis of the transformation theory are found to lead to best bound expressions. The simplicity and versatility of the transformation theory is emphasized.

### The Dislocation Formation Volume in Ice.

O. Bøcker Pedersen, presented at the International Symposium on Physics and Chemistry of Ice, Cambridge, September 1977. (Manuscript to be published in J. Glaciology).

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

### Brint i metal ved almindelig og høj temperature. (Hydrogen Embrittlement of Steel).

K. Rørbo, presented to Dansk Metallurgisk Selskab, Lyngby, May 1977. (Not available).

Damage of steel can take place as a consequence of contact with hydrogen both at elevated temperatures and at room temperature. At temperatures above 250°C a reaction between hydrogen diffusing into the steel and the cementite can take place, resulting in decarburization and crack formation. The problem can be counteracted by a certain content of carbide-forming elements as molybdenum and chromium as indicated in the Nelson diagram.

The hydrogen embrittlement seen at temperatures around room temperature is of a different nature and can result in delayed fracture of the steel at stresses significantly below the yield stress, but will not be revealed by a normal impact testing. This form of hydrogen embrittlement is of increasing significance in connection with the increased use of high-strength steel and high-purity hydrogen in modern technology. The mechanisms of the two sorts of hydrogen embrittlement are discussed and a number of practical examples are given.

## HVEM Studies of Void Formation in Cu-Ni Alloys.

B.N. Singh, T. Leffers and P. Barlow, presented at the 5th International Conference on High Voltage Electron Microscopy, Kyoto, August-September 1977. (Proceedings to be published).

The aim of the present research was to study the effect of alloying on the nucleation and growth behaviour of voids in Cu-Ni alloys. Thin foils of high-purity copper and Cu-Ni alloys were irradiated with 1 MeV electrons in a high voltage electron microscope. The experiments covered the composition range 0-10 wt.% Ni. Foils of the various compositions were irradiated at temperatures in the range 250-450°C.

The net effect of increasing Ni content was a drastic reduction in void swelling: at all irradiation temperatures the void swelling was reduced to practically zero when the Ni content reached 10%. The decrease in swelling is caused mainly by rapidly decreasing void growth rate with increasing Ni content. The void number density, on the other hand, tended to increase with Ni content.

The reduction in void swelling and growth rate caused by Ni additions is explained by vacancy or interstitial trapping at submicroscopic Ni precipitates. Our interpretation is based on experimental observations on the operation of climb sources in Cu-Ni alloys and on the climb rate of the dislocation loops emitted from these sources.

## Radiation Damage and Voids in Copper-Nickel Alloys.

B.N. Singh and T. Leffers, presented at Dansk Fysisk Selskabs forårsmøde, Helsingør, May 1977. (Not available).

The formation and growth of voids in copper-nickel alloys during 1 MeV electron irradiation was followed in an high voltage electron microscope. The experiments covered the composition range 0-10% Ni and the irradiation-temperature range 250-550°C.

For irradiation temperatures up to 450°C the void growth rate decreased with increasing Ni content for Ni contents above 1%. The void number density tended to increase slightly with Ni content, but the net effect of increasing Ni content was a drastic reduction in void swelling. For irradiation temperatures above 450°C the effect of Ni content on void behaviour was more complicated. However, for all irradiation temperatures it applied that void swelling was reduced to practically zero when the Ni content had reached 10%.

The reduction in void swelling and growth rate caused by Ni addition is explained by vacancy or interstitial trapping at submicroscopic Ni precipitates. According to the phase diagram there should be complete miscibility in the Cu-Ni system, and no precipitates are visible in the electron microscope, but the operation of special dislocation climb sources during irradiation proves the existence of the precipitates.

## Thermogravimetric Studies of Non-Stoichiometric Cerium Oxides under Isothermal and Quasi-Isothermal Conditions.

O. Toft Sørensen, presented at the 5th Scandinavian Symposium on Thermal Analysis, Trondheim, June 1977. (Manuscript to be published in J. Thermal Analysis).

Quasi-isothermal thermogravimetry is a new technique in which the programmed heating of a furnace automatically ceases when the rate of a reaction taking place in a sample, which is indicated by the DTG-signal, exceeds a preset limit. In this way reactions can be studied under nearly isothermal conditions. In this paper the data obtained using this method during oxidation and reduction experiments on non-stoichiometric cerium-oxides are compared with the data obtained by conventional isothermal thermogravimetry. The kinetics of the composition changes during isothermal reduction and oxidation of  $\text{CeO}_{2-x}$  are also analyzed. It appears that, with some reservations, the experimental data are best described by the rate equation for a diffusion-controlled reaction. Finally, both the isothermal and the quasi-isothermal data suggest that the  $\text{CeO}_{2-x}$  phase region in the phase diagram consists of several subphases, each with a characteristic activation energy for the diffusion-controlled reactions.

## Thermodynamic and Electron Microscopy Studies of Non-Stoichiometric Pu-oxides.

O. Toft Sørensen, L. Manes and I.L.F. Ray, presented at the American Ceramic Society Fall Meeting, Hyannis, September 1977. (Transcript available, 24 pp.).

Detailed thermodynamic studies of the Pu-oxides have indicated that the non-stoichiometric phase range consists of several subphases, each with a characteristic defect structure. The paper describes the analysis leading to this conclusion, and some electron-microscopy studies that were carried out to examine the structure of these subphases.

## Research on Zirconium hydriding and Palladium alloy - Hydrogen Systems at Risø National Laboratory.

B. Vigeholm, J. Kjøller, B. Larsen and O. Toft Sørensen, presented at the International Symposium on Metal Hydrides for Energy Storage, Geilo, August 1977. (Manuscript to be published in Int. J. of Hydrogen Energy).

In the course of contract research at our laboratory several investigations of metal-hydrogen systems have been carried out. The experience from these constitutes a major part of our research background for present work on metal hydrides for storage purposes.

This paper gives a short account of some of our findings which have not been published or reported superficially only in a different context. A procedure for hydriding zirconium and zirconium alloys is described and results from development of hydrogen-permeable palladium-boron alloys are presented.

## **Application of Metal-Hydrides in Energy Storage and Transport.**

**B. Vigeholm**, presented at Dansk Fysisk Selskabs forårsmøde, Helsingør, May 1977. (Not available).

Metal hydrides are potential carriers of hydrogen whether applied as a chemical raw material or as an energy vector. The fundamental property of the metal in this context is its ability to absorb hydrogen. Present solid state techniques permit some evaluation of this property. p-c-T correlations are calculated on basis of conduction-band theory and may take alloying effects into account. In applied materials research, this tool is hardly used. I shall describe the route normally taken in such a materials development programme and I shall suggest, how modern solid state physics might be of value in the process.

## **Hydrogen Storage and Transport in Mg-based Systems.**

**B. Vigeholm**, presented at an Ispra Course on the Hydrogen - Energy Concept, Ispra, May 1977. (Not available).

Independent of its potential as an energy vector hydrogen is a widely used chemical raw-material. As such it poses substantial problems in storage and transportation being extremely voluminous and volatile.

An economical alternative to conventional handling is the application of hydrides, particularly of Mg-based alloys.

We have investigated the case of larger scale consumption. Our conclusion is that Mg-based hydrides constitutes a potential cost saving means of transport.

The economical aspects and the technological problems involved will be described, together with an outlining of our present research programme.



STAFF OF THE DEPARTMENT

(as at 31 December, 1977)

Niels Hansen, head of department

Scientific staff

Adolph, E.  
Agerup, C.C.<sup>1)</sup>  
Andersen, Bjarne S.<sup>2)</sup>  
Bagger, C.  
Bilde-Sørensen, J.B.  
Borring, J.<sup>1)</sup>  
Brøndsted, P.  
Carlsen, Hans  
Christensen, Jørgen  
Debel, C.P.<sup>1)</sup>  
Domanus, J.  
Gundtoft, H.E.  
Hougaard, H.<sup>1)</sup>  
Jensen, Arne  
Johansen, Bjørn S.<sup>1)</sup>  
Kjær-Pedersen, N.  
Knudsen, Per  
Larsen, Niels  
Leffers, T.  
Lilholt, H.  
Lystrup, Aa.  
Nielsen, Arved  
Pedersen, A. Schrøder  
Pedersen, O. Bøcker<sup>2)</sup>  
Rasmussen, K. Viggo<sup>2)</sup>  
Rørbo, K.  
Singh, B.N.  
Stiff, J.  
Swindlehurst, W.E.  
Sørensen, O. Toft  
Vigeholm, B.

Technical staff

Adrian, F.  
Andersen, Axel<sup>1)</sup>, B.  
Andersen, Jan<sup>1)</sup>  
Arvidsson, E.W.  
Aukdal, J.A.  
Bülow-Christensen, C.J.  
Chabert, O.  
Cooper, D.  
Dreves, P.  
Feld, M.  
Frederiksen, Henning  
Friedrichsen, Uwe J.  
Hersbøll, B.  
Jensen, Børge

Jensen, Finn  
Jensen, Henning  
Jensen, Knud  
Jensen, Palle V.<sup>1)</sup>  
Jespersen, John<sup>1)</sup>  
Johansen, Claus  
Kjøller, J.  
Larsen, Bent  
Larsen, Jan  
Larsen, Kjeld J.C.  
Lindbo, J.  
Mikkelsen, Claus  
Mogensen, Erik B.  
Nielsen, Palle H.  
Nielsen, Torben  
Nilsson, H.  
Olesen, Preben B.  
Olsen, Arne  
Olsen, Benny  
Olsen, Bent  
Olsen, Niels H.  
Olsen, Ole  
Olsson, J.  
Pedersen, Børge  
Pedersen, Jørgen S.  
Petersen, Knud E.  
Paulsen, Henrik  
Poulsen, Einar R.V.  
Rasmussen, Allan E.  
Sandsted, K.<sup>1)</sup>  
Strauss, T.R.  
Svendsen, Bjarne  
Szøts, P.  
Sørensen, Erling<sup>1)</sup>  
Aagesen, Sven

Office staff

Christiansen, Grethe  
Eichen, A.M.  
Kristensen, Agnes  
Nielsen, Margit  
Olesen, Gyda  
van Zundert-Simonsen, M.

Consultants to the department

Non-destructive Testing

Niels Nielsen (Danish Welding  
Institute)

Physical Metallurgy

R.M.J. Cotterill (Technical  
University of Denmark)

G. Østberg (Technical University  
of Lund)

- 
- 1) Employee from the Elsinore Shipyard, Ltd.
  - 2) Post-graduate student from the Technical University of  
Denmark



**ISBN 87-550-0517-9**  
**ISSN 0418-6443**