From 22 June 1999 the Association started working on the Technology tasks assigned. Since then a number of changes have occurred which imposed re-organization of the work programme (i) a delay to the materials programme which was due to the long delay in the procurement of the SiC_f/SiC samples, (ii) the tasks' re-organisation approved by the Technology Subcommittee (the Hellenic Association's previous three tasks became one) and (iii) the approved Technology Work Programme 2000 for the Hellenic Association.

The work carried out in 1999 can be summarised as follows: (a) initial work towards the improvement of the irradiation facilities in order to increase the fast neutron flux, (b) construction of irradiation rings and tests, (c) experiments and studies on earlier generation SiC_f/SiC with emphasis on the effects of irradiation on their mechanical and physical properties in order to gain knowledge, develop and improve the techniques and prepare for the new materials. The SiSiC samples ordered from SEP were delivered in April 2000. This resulted in a delay in starting the work. However, very interesting results were obtained in as received N3-1 and N4-1 type specimens and specimens irradiated for 100 hrs. These results are presented in three reports and they have presented in conference at Frascati, Italy . Already specimens irradiated for longer times have come out of the reactor core and very soon will be investigated.

Other achievements for the period 1999-2000 are: (a) the installation of the new fast neutron irradiation facility at the "Demokritos" reactor (partially financed from UT, Technology programme and the Institute of Nuclear technology-Radiation Protection). This facility has improved the fast neutron flux by a factor of seven (7) and it is going to be mainly devoted for the European Fusion programme. This is an important development since the neutron irradiation facilities in Europe are diminishing. (b) The successful determination of the micromechanisms of SiC_f/SiC unirradiated and irradiated samples. (c) The neutron activation measurements of SiCSiC acted as a preliminary work for a technology task which we have been assigned for 2001 and (d) interesting results on SiC bond using SHS.

3a) UNDERLYING Technology

Fracture micromechanisms of SiC/SiC ceramic composites: Irradiation Effects on fracture and failure micro-mechanisms (1999-2000)

Although there has been some progress in understanding the irradiation effects on C/C composites no such knowledge exists for SiC/SiC materials. In particular, our knowledge in connecting irradiation damage, microstructural changes and mechanical and physical properties of such composites is lacking. This underlying technology project aims in understanding the mechanisms through which the irradiation damage degrades the mechanical and physical properties of the SiC_f/SiC composites and it is an integral part of our Technology programme (ADV1.1, ADV1.2 and ADV1.3 of the 1999 technology program and TTMA-001: SiC composites of the 2001 technology program). Improvements were made to the SEM jig for the in-situ SEM observations and first experiments were carried out using 2nd generation SiC/SiC. The results showed that R-curve is very shallow and the material fails before ultimate fracture toughness is reached (1999). SiC_f/SiC bar specimens types CERASEP N3-1 and CERASEP N4-1 were received from SNECMA in April 2000. The materials consist of a woven fiber perform of NICALON Si-C-O fibers in a SiC matrix produced by Chemical Vapour Infiltration (CVI). N3-1 uses the NICALON-S grade fibers (Structural grade) whereas N4-1 uses the Hi-NICALON fiber grade. Neutron Irradiation was carried out at the Experimental Reactor at the Institute of Nuclear Technology and Radiation protection of NCSR "Demokritos". At present, results are available for neutron irradiation up to 100 hrs (fast neutron fluence of 1.6×10^{19} n/cm^2). The materials were characterized by measurement of their Toughness, Energy dissipation and R-curve behaviour and in-situ fracture observations to ascertain changes in fracture micromechanisms before and after neutron irradiation.

The main conclusion are:

- Crack arrest after fracture was found to be about 40% of MOR in N3-1 but significantly lower in N4.
- The materials display moderate toughness and R-curves: in bending tests, Kic of N3-1 increased from about 8 to 12 $Mpam^{-1/2}$ over a crack extension of about 1 mm. However, Kic for N4-1 ranged from 7 to 9 $Mpam^{-1/2}$ over 1 mm.
- Energy dissipation was moderate in N3-1 but low in N4-1. Mechanisms for energy dissipation include elastic deformation of fiber structure, matrix cracking, fiber bundle bending and fracture and fiber pull-out. In N4-1 fiber bundle fracture appears to dominate, whereas in N3-1 fiber pull-out dominates.
- Irradiation to a fast neutron fluence of 1.6×10^{19} n/cm^2 does not affect the strength, density and porosity of the materials, but it has a strong effect on the energy dissipation and

micromechanisms of fracture.

- Toughness of irradiated materials is substantially reduced and R-curves in irradiated materials are lower and shallower indicating lower activity of fracture micromechanisms.

- Irradiation appears to change the nature of the matrix-fiber interface and fibre pull-out in both materials is significantly suppressed after irradiation.

The work is continuing with longer irradiation periods.

Preliminary evaluation of Impurities and Induced Activities of SiC composites irradiated at "Demokritos" reactor (1999–2000)

The evaluation of impurities and induced activities of SiC composites irradiated at the fast neutron irradiation facility developed at "Demokritos" researched reactor for the requirements of the EURADOS fusion program is of importance for radiation protection and safety purposes. Instrumental Neutron Activation Analysis (INAA) was applied in order to determine the levels of impurities and consequently to evaluate the induced activity at selected samples prior to the beginning of long-term irradiation cycles. The results of the measurements indicated peaks attributed to the radionuclides ^{182}Ta, ^{51}Cr, ^{110m}Ag, ^{124}Sb, ^{58}Co, ^{54}Mn, ^{59}Fe, ^{65}Zn, ^{22}Na, ^{60}Co. However, the results of preliminary calculations indicated that dose-rate levels post irradiation would be within the safety margins set by the health physics office.

New fast neutron irradiation facility (1999)

In order to successfully carry out the fusion Technology and Underlying Technology program, which requires sample irradiation at a fast neutron fluence, it was necessary to modify "Demokritos" research reactor irradiation facilities. Normally, in a Research Reactor for isotope production and other applications the main activities are directed towards irradiation at thermal neutron fluxes. Consequently, the available local expertise was involved to the effect. Monte Carlo neutron transport calculations were performed using code MCNP-4B in order to propose the required core modifications to develop an in-core fast neutron irradiation facility, complying to the fusion program irradiation requirements. Several different core arrangements were investigated and an optimum irradiation facility design was proposed. By the end of 1999 it was apparent that a considerable gain in the fast neutron flux of a factor of seven was at hand, while a useful irradiation volume of about 500 cm^3 could be obtained. Moreover, the problem of air to water reactivity worth of the proposed facility was examined in connection with accidental

ingress of pool water in the irradiation tube/container. All critically safety parameters examined were found to be well within the safety margins. Furthermore, preliminary neutron fluence measurements using foil activation were performed in order to verify the results of the calculation. We stress that a very good agreement between calculations and measurements was observed. A detailed determination of the neutron fluence spectrum at the in-core fast neutron irradiation facility using a multi-foil activation-iterative technique will be performed during the second year of the program. (See also
Annex XIV
and
Annex XV
.)

Irradiation rings and irradiation procedure (1999)

The long irradiation periods required for the fusion programme as well as the intense fast-neutron fluxes, necessitated the design, construction and testing of specialised specimen support rings. Testing of the new rings has now been completed successfully. A new rig development for the irradiation of a large number of samples and of better remaining activity is under development. Also it was carried out work for the definition of the appropriate procedure for in-core sample placing and recovering in compliance with the radiation protection regulations.

Monte Carlo simulation studies related to the development of a fast neutron irradiation facility at "Demokritos" research reactor (2000)

Monte Carlo neutron transport code MCNP-4B was used to model GRR1 "Demokritos" research reactor core configuration. Scope of the study was the design of a fast neutron irradiation facility for the requirements of the Euratom fusion program. Design considerations included maximization of fast neutron flux and criticality safety. The results of the study indicated that the development of a vertical access in-core irradiation trap at lattice position D-4 was necessary, since such a facility would fully exploit the maximum of fast neutron flux distribution near the reactor core geometrical center. The problem of air to water reactivity worth of the proposed irradiation trap in connection with accidental ingress of pool water into the tube was investigated. Criticality calculations performed using the MCNP code suggested an increase in reactivity of 11c. Detailed reactor kinetics and thermal-hydraulic analysis performed using the PARET code indicated that the variations of reactor power and clad temperature due to this reactivity insertion are well within the safety margins, even with simultaneous loss of one primary pumps. Consequently, a detailed MCNP model of the final irradiation facility was performed in order to characterize the neutron field properties as related to the requirements of the material irradiation tests. The results of the simulation indicated that the maximum fast neutron fluence rate at D-4 was of abut 4.5×10^{13} $cm^{-2}s^{-1}$ at 5 MW, with a fast to total neutron flux ration of about 0.4. Moreover, the effective irradiation volume was of about 125 cm^3 within a fast neutron flux uniformity of better than ±5%; or, a total irradiation volume of 505 cm^3 defined by a 50% reduction of the peak fast neutron flux. Further advantages of the design were the simplicity of vertical in-core sample insertion and overall radiation safety to reactor operating personnel. (See also Annex XVI.)

3b. TECHNOLOGY

Improvement of irradiation resistance of SiCSiC-Mechanical properties (1999)

Work was carried out on previous generation plain weave SiC_f/SiC with non-stoichiometric (SiO_2-C-Si) Nicalon fibres. Preliminary irradiation tests showed fibre swell resulting in extensive matrix cracking and overall damage to the structure. The interfacial bonding is degraded and toughening decreases substantially. The SEM observations show that toughening mechanisms are ineffective. Low mechanical strength is the overall result. These results are believed to be due to the low grade non-stoichiometric Nicalon fibres used in those early materials. The new materials are made with more advanced Nicalon fibres and are expected to show improved irradiation resistance. During 2000 physical and mechanical characterisation to the N3-1 and N4-1 CERASEP SiC_f/SiC samples will be carried out including density and porosity, bending strength, structure and morphology before and after irradiation.

Bonding of SiC_f/SiC (1999)

Joining of SiC is usually accomplished by brazing in neutral atmosphere of vacuum and ambient temperatures above 1300°C using specially develop brazing alloys. In the meeting of the working group on SiC_f/SiC it was decided that the Hellenic Association's contribution would be in the development of a new more advantageous technique based on the Wave Combustion Bonding (WCB) in which we have expertise. This new joining method creates a SiC bonding layer by in-situ combustion synthesis. Combustion takes place at Tc>2500°C and bonding tasks place by localized melting and diffusion. The whole process last only a few seconds at an ambient temperature of about 1000°C. Work up to now has concentrated on the development of the synthesis process via a number of different routes (different powder mixtures and conditions) and preliminary tests on SiC specimens. During 2000 we will further develop this method by application to the new N3-1 and N4-1 materials. Irradiation tests will also be carried out. (See also Annex XVII .)

Irradiation resistance of SiCSiC and effects on mechanical properties (2000)

SiC_f/SiC bar types CERASEP N3-1 and CERASEP N4-1 were received from SNECMA in April 2000. The materials consist of a woven fibre perform of NICALON Si-C-O fibers in SiC matrix produced by Chemical Vapour Infiltration (CVI). N3-1 uses the NICALON –S grade fibers (Structural grade) whereas N4-1 uses the Hi-NICALON fiber grade.)

Neutron Irradiation was carried out at the Experimental Reactor at the Institute of Nuclear Technology and Radiation protection of NCSR "Demokritos". At present results are available for effects of 100 hrs (fast neutron fluence of 1.6×10^{19} n/cm^2).

The materials were characterized using ceramography, densitometry, factography and by measurement of their elastic and bulk mechanical properties before and after neutron irradiation and by measurement of their Toughness, Energy dissipation and R-curve behaviour and by in-situ SEM fracture observations to ascertain changes in fracture micromechanisms.

The density, open and total porosity, Young's modulus (in bending), initial matrix cracking and bending strength (MOR) of the SiCf/SiC composites tested in this work (CERASEP N3-1 and N4-1) are not affected significantly by neutron irradiation up to a fast neutron fluence of 1.6×10^{19} n/cm^2. The work is continuing with neutron irradiations planned for longer periods and at higher fast neutron fluences. (See also Annex XVIII .)

Joining of SiCSiC using a controlled wave combustion method

Effective joining of SiC_f/SiC for potential use on the plasma-face blanket of a fusion reactor must satisfy a number of criteria:

- The joint material must be chemically and physically compatible with SiC_f/SiC.
- The joints must have adequate mechanical and physical properties and no open porosity.
- The method used must not adversely affect the properties of the materials being joined.
- The method used must enable joining of relatively large areas both between segments of SiC_f/SiC and with the backing material.

We have used the Self-Propagating High-Temperature Combustion Synthesis (SHS) method for synthesizing materials in-situ between the faces of SiC specimens thereby enabling joining.

The current project aims at firstly finding the right composition of the starting materials and processing parameters for the in-situ production of SiC at as low temperatures as possible and secondly to demonstrate the possibility of joining of SiC using wave combustion.

Experiments were carried out with range of pre-cursors and initiation conditions and the synthesized materials were characterized by a X-ray diffraction, measurement of their density and porosity and by microscopy. In conclusion: (a) the SHS method was used to produce SiC from a number of systems over very short times by the propagation of a combustion wave, (b) this "wave combustion" method appears to offer the potential for in-situ synthesis and joining of SiC materials and (c) the joints are being characterized by measurement of their physical and their mechanical properties.

The high porosity of the joints and the reproducibility of the wetting behavior with monolithic or composite SiC materials is being investigated further. (See also Annex XIX .)

Through-thickness mechanical properties of SNECMA CERASEO SiCSIC composites

Effective modelling of mechanical behavior of plasma-facing components of the fusion reactor blanket require a good knowledge of the in through-thickness properties of the material. This report presents the results obtained to-date from measurements of through-thickness elastic and mechanical properties. These properties can be used as input for modelling components and for developing accurate mock-ups of various components of the blanket.

SiC_f/SiC bar specimens types CERASEP N3-1 and CERASEP N4-1 were received from SNECMA. The materials have been characterized using ceramography (optical and electron microscopy) and by measurement of their through-thickness mechanical and elastic properties using specially designed specimen configurations and rings. The main conclusion are: (a) The through-thickness properties are lower than those in plane and failure takes place at lower strength and (b) the transverse plies (through-thickness) of the fibres are not sufficient to offer isotropic properties to the material.