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Summary of the 7th International Workshop on Spallation Materials Technology (IWSMT-7)

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As in earlier meetings of this series, IWSMT-7 was aimed at providing a forum for demonstrating the progress and achievements in new and existing spallation neutron sources and ADS projects, and at presenting the latest results from materials related research. These topics included effects of radiation damage and transmutation products (especially helium and hydrogen) in structural materials, pressure wave induced cavitation erosion effects in liquid mercury targets and compatibility of liquid lead-bismuth eutectic (LBE) with structural materials in the representative conditions of ADS liquid metal targets. The discussion periods focused on open questions encountered in developing high power spallation targets. The objectives of the meeting were reached by the 52 presentations on different topics. This summary presents the information mainly from the papers included in these proceedings. Most of the presentations are published as full papers in these proceedings. Interesting results from some of those presentations that could not be published for various reasons are also included in this summary.

The workshop began with overviews of high power spallation neutron sources that are presently under construction, being planned, or in operation. Mansur and Haines described the status of the Spallation Neutron Source (SNS), and then focused on the materials work for the liquid mercury target. The initial concept planning began in 1994; the facility is expected to achieve first beam on target in June 2006. Neutron scattering research is the primary purpose of the facility. In addition there is significant development planned in target technology, materials and instruments. The detailed design of the target, inner reflector region and moderators was presented. The target is liquid mercury contained in a four walled target module, consisting of a double-walled inner mercury target and a double-walled outer water-cooled shroud. Radiation effects, cavitation erosion and compatibility studies, which have been the main components of the R&D program, were reviewed and their highlights described. Near term future work will concentrate mainly on cavitation erosion.

Groeschel et al. reviewed the design and fabrication progress of the MEGAPIE facility, which is to be installed at the SINQ. This device is a liquid metal (lead-bismuth eutectic) target and support system, which will serve as a neutron production target and at the same time demonstrate the performance and help develop the technology for liquid metal targets relevant to ADS. It is expected to begin operation in May 2006. Descriptions of

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experience with fabrication of the various components of the system made up the major part of this presentation. Radiation effects issues, safety considerations and reliability predictions were covered. It was emphasized that comprehensive non-destructive testing must be applied to facilities of this type to detect manufacturing defects and other potential issues that must be corrected prior to operation.

Kikuchi et al. reviewed the status of the Japan Proton Accelerator Research Complex (J-PARC). In April of 2001, the two separate designs for a high power accelerator put forth by JAEA and KEK were unified and construction began. The facility will eventually contain four different experimental areas for materials and life sciences, nuclear and particle physics, neutrino studies and nuclear transmutation studies. The spallation neutron source of the materials and life sciences experimental area is of somewhat similar design to the SNS, and is expected to begin operation in 2007. Materials R&D was described for this part of the facility and also for the muon production target. Kawai et al. described the development of materials for resistance to beam impact and radiation damage. Developments of new materials and processing techniques were highlighted, including grain boundary engineering and mechanical alloying, which hold promise for better performance.

Maloy et al. described the conceptual design for a new Materials Test Station (MTS), which has been proposed for installation in the beam dump area of the LANSCE facility. The facility would utilize a vacuum environment design, rather than air to avoid radioactive emissions. The proposed spallation target consists of plates of tungsten clad with tantalum or with stainless steel to prevent corrosion by the water coolant. The design is optimized to obtain a neutron spectrum similar to that of a fast reactor, although the helium production will be higher than in a fast reactor, as a consequence of the high energy particles in the neutron spectrum. Anticipated materials issues for fabrication of the facility were described. It is expected that it could be completed on a four year schedule, once approval for the start of work is obtained. It is expected that an upgrade of the LANSCE facility would be accomplished at the same time.

Dai et al. described the progress of the SINQ target irradiation program (STIP). This has been a productive program with a number of collaborating institutions from around the world. It has made available a large number of specimens exposed to various combinations of displacement dose and temperature in a spallation radiation damage spectrum. Irradiations of the experiments designated STIP I, II, III have been completed. STIP IV was in progress and STIP V was in the planning and specimen preparation stages. The focus of STIP III was on the alloys T91, Eurofer 97, F82H and 316L. The performance of the Al–Mg alloy window was described along with examples of doses and transmutation gas accumulation calculations for the specimens. For example, a typical specimen received 10.5 dpa, 958 appm He and 5813 appm H. In this experiment temperatures were measured for specimens up to 500 °C.

As one of the main topics, the effects of irradiation and He and H on the mechanical properties of structural materials were discussed in ten talks. In the invited presentation, Odette et al. collected and analyzed a variety of data of ferritic/martensitic steels (FMS) pertinent to the issue of the effect of helium on fast fracture by evaluating the relation between DBTT shift (ΔT_c) and hardening ($\Delta\sigma_y$). The results suggest that up to concentrations several hundred appm, helium has little effect on ΔT_c. However, at higher concentrations the data is consistent with the hypothesis that accumulation of helium weakens grain boundaries to the point where a non-hardening-embrittlement effect emerges as signaled by both higher ΔT_c and inter-granular fracture.

In two other talks, the results of bending and tensile tests on specimens of FMS irradiated in STIP-I and -II were presented. Jia and Dai showed that the fracture toughness of FMS specimens irradiated in STIP-I to doses up to 9 dpa in 100–250 °C decreased with increasing irradiation dose. Specimens tested at irradiation temperatures of 250 °C indicated that the fracture toughness of all three FMS (F82H, Optifer-V and T91) remained above 100 MPa m$^{1/2}$. But one T91 specimen of 4.3 dpa tested at room temperature was totally brittle and failed in the elastic deformation region. Maloy et al. measured the tensile properties of FMS EP823 and HT-9 at 25 °C, 250 °C, and 400 °C after irradiation in STIP II to doses up to 20 dpa at temperatures up to 350 °C. The losses in ductility concomitant with increases in yield stress were observed in both alloys, although the embrittlement was more severe in the EP-823. EP-823 specimens broke in the elastic regime at room temperature after irradiation. The yield stress also increased more steeply with dose in EP-823. These results suggest a strong effect of silicon on mechanical properties of EP-823.

Toloczko et al. showed tensile results of FMS HT-9 and JFMS irradiated in FFTF to up to $\sim$ 70 dpa at 373–433 °C and tested at 25 °C and 400 °C. The results indicate that the radiation hardening decreases with increasing irradiation temperature from 373 °C to 473 °C. The JFMS alloy, which has 0.7 wt % silicon, exhibits approximately a factor of two increase in yield strength between tests at 427 °C and at 373 °C, and shows an increase in hardening with increasing dose. A comparison of the JFMS tensile properties to the properties of HT-9 suggests that this hardening is due to precipitation of a Si-rich Laves phase in this alloy. The HT-9 alloy shows hardening during irradiation at 373 °C, but less for irradiations at 427 °C and no increase with increasing dose beyond 10 dpa.

An analysis of the tensile response and microstructural evolution of selected series of unirradiated and irradiated 316L tested at different temperatures was carried out by Stubbins et al. to establish the concept of critical stress for plastic instability and identify the controlling mechanism of flow localization. The main conclusions were: (1) irradiated and unirradiated 316L suffered flow localization when the irradiation-induced hardening increased the yield strength to the level of the critical stress which was not a function of irradiation level, (2) it was found that twinning was an important deformation mechanism at lower temperatures but was not at above about 200 °C where the stress to activate twinning became excessively high, which limited the deformation and led to the flow localization process.

He effects in FMS were studied in three ion implantation experiments. Henry et al. performed bending tests at room temperature on T91 miniature Charpy specimens implanted at 250 °C in the notch with 0.25 at.% helium. 'Pop-in' phenomena were systematically observed and SEM observations revealed a fully brittle fracture appearance in the implanted zones. In addition, finite-element (FE) simulations of the tests performed on unimplanted and implanted specimens were also carried out to determine stress and strain fields at the onset of crack propagation. The FE simulation predicted a lower fracture toughness value for T91 implanted with 0.25 at.% helium at room temperature than that of unimplanted steel measured at −170 °C. In another experiment, Jung et al. investigated EUROFER97 which was homogeneously implanted at 250 °C with 0.25 at.% helium. Tensile testing at room temperature and 250 °C showed increase in strength and decrease of ductility by the implantation. Both changes recovered during annealing (10 h at 550 °C or 750 °C): while the strength was reduced by annealing to values between unimplanted and implanted condition, ductility was increased even beyond that of virgin material. He effects were also studied by Wakai et al. using different techniques such as triple-beam irradiation and B-doped material irradiated with neutrons and producing helium by (n, alpha) reactions. Micro-hardness and tensile results showed greater hardening in specimens with higher He concentrations. In addition, it was found that the swelling in dual-beam (He + Fe) irradiated specimens exhibited a peak at 430 °C.

H effects on tensile properties of FMS and 304L were investigated by Toloczko et al. using H charged at elevated temperature and high pressure. The results demonstrated that the measured H contents in 304L were inline with the predicted values, while the H contents in FMS were below the detection limit. Tensile tests showed that up to 2200 appm H did not affect the ductility but induced slight hardening in 304L.

The microstructure of FMS irradiated in STIP was studied by Jia and Dai using TEM and by Grosse et al. with SANS and ASAXS (please spell out this acronym) analyses. TEM observations were performed on F82H specimens irradiated to doses up to 20 dpa at temperatures up to $\sim$ 400 °C. The results illustrated that He bubbles became much larger in specimens irradiated to 18 dpa with $\sim$1600 appm He at 350 °C. In a specimen irradiated to 20 dpa with 1800 appm He at 400 °C, voids of up to 50 nm diameter were observed accompanied by a high-density of small bubbles of 2 or 3 nm diameter. In the SANS experiments strong effect of irradiation was found. The volume fraction and size of the radiation defects detected by SANS did not change much with fluence. The ASAXS investigations indicated that the radiation defects detected by SANS were not Cr-rich small precipitates. For austenitic steels, Sawai et al. made TEM observations on 316F and JPCA irradiated in STIP up to 10 dpa at 300 °C and below. In the 316F specimen irradiated at 300 °C, Frank loops up to 30 nm and stacking fault tetrahedra of about 2 nm were detected. The features in both steels were very similar.

Vontobel et al. inspected some irradiated specimen rods of STIP using the neutron radiography technique. It was shown that this method was very useful for non-destructive analysis of irradiated materials such as the irradiated specimen rods of

STIP and, in particularly, detecting hydrides formed in irradiated Zircaloys.

Gas (He, H, D etc) production, retention and release continue to be important topics in this series meeting. A number of authors presented a large amount of interesting results. Garner et al. showed that under certain conditions, hydrogen can be stored in irradiated nickel and stainless steels at levels strongly in excess of that predicted by Sieverts Law. These conditions are first, the availability of hydrogen from various radiolytic and environmental sources and second, the formation of radiation-induced cavities to store hydrogen. These cavities can be highly pressurized bubbles or under-pressurized voids, with helium in the cavities at either low or very high levels. Transmutant sources of hydrogen are often insufficient to pressurize these cavities, and therefore environmental sources are required. Tolstolutskaya et al. also found that retention of hydrogen and deuterium is strongly enhanced by the presence of large amounts of helium and lattice damage. In the TDS (thermal-desorption-spectroscopy) analyses on EC 316LN, F82H, $AlMg_3$ and Zircaloy-2 irradiated in STIP Oliver et al. noticed that He and H release measurements showed considerable levels of D T (deuterium and tritium) species which generally mirrored those of hydrogen. H release occurred from about 300 °C for the $AlMg_3$ to about 800 °C for the Zircaloy-2. For the Zircaloy-2 and the steels, He release began to occur at between 1100 °C and 1200 °C. Similarly, Kikuchi et al. found that the release of tritium began at temperatures over 250 °C in steels irradiated in STIP. The ratio of residual to generated tritium is estimated to be less than 20%.

To develop high-power liquid metal spallation targets, studies on liquid metal induced corrosion, erosion and embrittlement effects in structural materials have received special attention in the last few years. A significant number of new results were reported at the meeting.

For the liquid mercury targets, pitting damage in the beam window induced by cavitation is the issue of most concern and is being investigated intensively in the SNS and J-PARC projects. Futakawa et al. observed that the pitting damage formation in SA316 and CW316KL at impact cycles up to 10 million could be divided into three phases: Phase 1, isolated individual pits were formed up to 10^4 cycles; Phase 2, pits overlap and the fraction of eroded area approached 1 between 10^5 and 10^6 cycles, and; Phase 3, homogeneous erosion with mass loss started between 10^6 and 10^7 cycles. They also noticed that in the incubation period, the fatigue limit was hardly affected by the pitting; in the steady state, the pitting damage degraded the fatigue limit. The incubation period of CW316KL was longer than that of SA316, while degradation rate of the fatigue limit due to pitting damage in steady state was higher in CW316KL than in SA316.

Although it has been shown that surface hardening treatments can play an important role in mitigating the threat of the pitting erosion, the behaviors of the hardened surface layer under irradiation had not been reported prior to the present meeting. Farrell and Byun tested annealed and 20% cold-rolled 316LN steel, with and without a carburized solid solution layer near the surface, after neutron irradiation to 1 dpa at 60–100 °C. It was observed that the hardness of the layers was increased by 2–12%, compared to increases of 81% and 43% for the annealed and 20% cold rolled non-carburized control materials, respectively. Optical microscopy examinations of the surfaces of the as-carburized-and-irradiated specimens revealed no sign of decomposition attributable to irradiation.

To solve the problem of cavitation erosion by manipulating the mercury rather than the structural materials, Thomsen suggested a number of ideas to protect a solid surface. These focused on ensuring a sufficient density of gas bubbles of suitable composition and size residing right at the mercury–steel interface and its close vicinity.

For developing liquid lead or lead-bismuth targets, the compatibility of lead or lead-bismuth with structural materials is also of great concern. Recent studies showed that the corrosion of steels in lead-bismuth eutectic (LBE) can be severe at temperatures above 400 °C and oxygen content in LBE plays an important role in corrosion mechanisms.

Schroer et al. reported the results of exposure experiments on T91 for 1200, 2998 and 5016 h. At 550 °C, oxidation was the only mode of material degradation for T91 in flowing LBE (2 m/s) when the oxygen activity corresponded to $10^{-4} \leqslant a_{PbO} \leqslant 10^{-2}$. The mean oxidation rate in the course of the first 1200 h approximates 0.13 mm/y. Lower oxygen activity in the LBE ($10^{-6} \leqslant a_{PbO} \leqslant 10^{-4}$) may be beneficial in respect to the oxidation rate, but undershooting a certain value around the threshold for Fe_3O_4 stability ($a_{PbO} = 10^{-5.2}$) poses the risk of liquid–metal attack accompanied by high local material consumption.All things considered, T91 showed promising (short-term) oxidation behaviour in flowing LBE at 550 °C and $10^{-4} \leqslant a_{PbO} \leqslant 10^{-2}$ with respect to the suppression of liquid–metal attack.

In the corrosion tests on 316L in flowing LBE at low oxygen concentration of $10^{-8} \sim 10^{-10}$wt%, Benamati et al. studied the incubation period of the corrosion process under selected conditions. It was observed that after 1000 h of exposure, a corrosion process based on elemental dissolution started. But in only a few areas the LBE penetrated into the steel matrix, down to a maximum of 5 μm, without preferential paths. In another experiment performed at 550 °C, Kondo and Takahashi found that Al- and Si-rich steels showed no obvious up to 2000 h exposure in flowing LBE.

Although the irradiation time was not long, LiSoR experiments obtained the first experience on LBE corrosion under intensive proton irradiation. Glasbrenner et al. reported the observations of the LiSoR-3 experiment, in which a small area of the T91 tube in a LBE loop was exposed to 72 MeV protons for about 260 h. The averaged beam current was about 20 μA/cm^2 and the maximum irradiation dose was about 0.2 dpa. SIMS and SEM/EDX investigations showed no corrosion attack of LBE to the steel, and no micro cracks formed. An oxide layer consisting of a duplex structure was formed on the steel surfaces.

Glasbrenner and Groeschel also performed tests on different coatings in a LBE loop. Three types of coating TiN + 2–3% Cr (CVD), CrN + W (PVD) and DLC (diamond like carbon) were studied. Coated T91 specimens were exposed to flowing LBE at 350 °C and a static stress (0, 70, 150 and 200 MPa) for 1000, 3000 and 6000 h of exposure. The SEM and EDX analyses showed excellent results from all three type coatings concerning their compatibility in LBE. The static stress influenced the stability of CrN and DLC coating but not the TiN coating.

Although the corrosion rate is low at temperatures below 400 °C, the embrittling effect of LBE on ferritic/martensitic steels can be very pronounced. Dai et al. performed slow-strain-rate tensile (SSRT) tests on T91 specimens in static LBE in the temperature range of 250–425°C. Strong embrittlement behavior reflected by significant reduction of ductility was observed in tests on those specimens with microcracks on the lateral surfaces at temperatures ⩾300 °C, while no embrittlement behavior was seen in the tests on specimens without surface microcracks. The yield and ultimate tensile strengths and uniform elongation were not affected, though the total elongation decreased substantially. SEM observations showed that the specimens ruptured in a brittle fracture mode when the embrittlement behavior appeared. The results indicated that surface cracks or flaws play an important role in the LBE embrittlement behavior. In the tensile tests on the T91 specimens cut from the test sections of LiSoR-2 to -4, it was shown that the level of embrittlement was affected by the tensile strain rate. However, when LiSoR irradiated specimens were tested in Ar gas, no large reduction in elongation was observed, which suggested that the synergistic embrittlement effects of irradiation and LBE were not evident in LiSoR irradiation to a low dose of 0.2 dpa.

In SCK/CEN (Belgium) a set of tensile tests on T91 and 316L specimens in unirradiated condition or irradiated to about 1.5 dpa were carried out in both static LBE and Ar gas. The specimens were prepared with relatively smooth machined surfaces. The unirradiated T91 specimens were tested in the temperature range between 150 °C and 450 °C at strain rates between 1×10^{-3} and 1×10^{-6}. No embrittlement or decrease in mechanical properties by LBE was observed. However, where a sufficiently large surface defect such as a local corrosion attack or a notch was present, the total elongation was reduced when tested in LBE. Similarly, the tests at 200 °C on T91 and 316L specimens irradiated up to 1.5 dpa did not show LBE embrittlement either. These results indicate again that the surface condition is important for the occurrence of embrittlement in LBE.

As for of heavy liquid metal technology, Foletti et al. presented an overview of the R&D activities carried out in ENEA, Italy. Kikuchi et al. reported the results of measurements of flow velocity profile of LBE using the ultrasonic Doppler velocity profiler (UVDP) technique.

In the last two sessions, some results from neutronics calculations and target development fields were presented.

An upgraded radiation damage database was described by Lu et al. The database contains proton and neutron cross sections for production of damage energy, displacements, helium, hydrogen, and heavier transmutation products. The targets in the database include 23 elements from Mg to U and eight practical alloys, but only Al, Fe, and W were discussed in the paper. Damage energy cross sections were presented for 20–3200 MeV protons based on intranuclear cascade (INC) models (Bertini, CEM2k, and ISABEL) and for 1–2000 MeV protons based on classical Rutherford scattering and SRIM. These cross sections were used to calculate displacement production in STIP-III. In another work, Vladimirov and Möslang evaluated irradiation conditions of the ADS beam window using Monte Carlo neutron, photon and charged particle transport code MCNPX. A realistic simulation was performed for the European experimental ADS (XADS) facility to understand the spallation process and transport of generated nucleons as well as evaluation of various damage and operational characteristics like displacement damage, heat deposition, and gas and spallation element production rates.

To develop Ag–In–Cd alloy decoupler, a method is needed to bond the Al alloy (Al5083) and the neutron absorber, the ternary Ag–In–Cd alloy. Teshigawara et al. found that an optimum HIP condition was obtained at 803 K and 100 MPa with a hold for 10 min for small test pieces (22 mm in dia. $\times$ 6 mm in height). A hardened layer due to the formation of $AlAg_2$ was found in the bonding layer. However, the rupture strength of the bonding layer was higher than 30 MPa, the calculated design stress. Bonding tests of a large size piece ($200 \times 200 \times 30$ mm^3), which simulated the real scale also produced a good result.

Schuumans et al. presented the main features of an experimental ADS, MYRRHA, under developed at SCK, Belgium. Besides the design of the different components, the main outcomes of the materials R&D were demonstrated. Based on the existing database, T91 was considered as the primary candidate for the fuel cladding and target structural materials and 316L for the vessel and ancillary system.

Last but not least, Dai et al. used the up-dated knowledge of radiation damage and LBE embrittlement to evaluate the expected lifetime of the MEGAPIE target beam window. The DBTT data of martensitic steels irradiated in STIP and fracture toughness values of T91 specimens tested in contact with LBE suggest a lower bound of the lifetime of the T91 beam window to be limited to a dose of about 6 dpa, when the steel becomes brittle at the lowest operation temperature, 230 °C, and with a safety margin of 30%.