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R&D IN ZIRCONIUM TECHNOLOGY AT BARC

As most of you are aware, zirconium technology is firmly established in India, based on the results of the concerted efforts over the last four decades at various units of DAE. With a sense of pride, I recall the days in the early sixties when the first success in separating hafnium from zirconium by solvent extraction, the first production of zirconium sponge by Kroll process and the first hot extrusion of zirconium tubular products were achieved in this Centre. There was tremendous excitement in those days for putting India amongst the group of very few nations who had mastered this technology. Immediately thereafter, the chemical engineers and metallurgists of this Centre joined hands in optimising the parameters of the various unit operations and designing a larger plant for producing zirconium metal from the indigenously available zircon sand. The technology thus developed was subsequently transferred to NFC who took up the challenge of regular production of zirconium alloy components to meet the requirement of all the nuclear power stations of the country. As we are aware, a 220 MW(e) PHWR unit needs initially 12.3 MT of Zr – 2.5 Nb alloy (for pressure tubes) along with a total of 10.7 MT of Zircaloy-4 (5.2 MT for calandria tubes and 5.5 MT for fuel clad) while the corresponding figures for a 500 MW(e) PHWR system are 30 MT, 9.7 MT and 12.4

(Lecture delivered by Mr B. Bhattacharjee, Director, BARC, at the inaugural session of the Symposium ' Zirconium-2002 ', held at BARC during September 11-13, 2002)

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MT respectively. Besides, a PHWR also calls for a sizeable requirement of these alloys periodically and this task is presently being executed at NFC with admirable competence.

To this audience, it may be superfluous to mention that alloys of zirconium are the most important structural materials within the core of nuclear reactors. Owing to the combination of a variety of properties such as (i) low thermal neutron absorption cross section, (ii) adequate strength and ductility at reactor operating temperatures, (iii) good corrosion resistance at high temperatures in aqueous environments, (iv) reasonable dimensional stability under irradiation; and (v) good compatibility with the fuel material, zirconium alloys have been a natural choice for fuel cladding in PWRs and BWRs to start with.

When the pressure tube design for reactors like PHWR came into being, again there was no other choice but to accept zirconium alloys as the pressure tube materials for reactors fuelled with natural uranium. We must remember that during those days long term mechanical property data for zirconium alloys were not available and there were no codes concerning the use of such materials in pressure boundary applications. It was indeed a bold decision to adopt a new material with somewhat uncertain long term behaviour for such a critical application. Today, after the successful operation of the pressure tube type reactors in several countries of the world, we gratefully acknowledge the sagacity of that decision.

In recent times, a more efficient process has been developed for the separation of zirconium and hafnium. This process is based on a pyro-metallurgical route involving extractive distillation of a molten salt of Zr (Hf) chloride. It is satisfying to note that this process, which was initially developed in the bench scale in BARC, has now been adopted in NFC in a pilot plant scale for a detailed techno-economic assessment of the process.

The history of zirconium alloy development is quite interesting. As we are aware, the consideration of neutron absorption property restricts the number of alloying elements which can be added to zirconium. Since the starting material for alloy making is Kroll zirconium, which is invariably contaminated with nitrogen to a level of about 50 ppm, it was necessary to add an alloying element which can take care of the deleterious effect of nitrogen on the corrosion resistance of zirconium alloys in aqueous environments. Tin has been the natural choice as it has a low neutron absorption cross section and it can complex with nitrogen, thereby scavenging nitrogen out of the zirconium lattice. This was the rationale behind the development of Zircaloy-1 (Zr-2.5% Sn). The development of Zircaloy-2 (Zr – 1.5 Sn – 0.2 Fe – 0.1 Cr – 0.05 Ni) which, as we know, has been the work horse material for nuclear core components world wide, came about by accidental addition of a piece of stainless steel to a Zicaloy-1 melt. As the story goes, an innovative melter tried to improve upon the corrosion resistance of zircaloy by adding stainless steel, the established corrosion resistant alloy. This serendipitous discovery has dramatically influenced the nuclear industry in general and zirconium technology in particular in a very significant manner. In Zircaloy-4 (Zr – 1.5 Sn – 0.2 Fe – 0.1 Cr), nickel has been removed (retaining tin, iron and chromium). Later on, zirconium-niobium alloy (Zr – 2.5 Nb) has replaced Zircaloy-2 for pressure tubes in PHWRs because of its better strength and irradiation creep properties. Today, however, there is a rethinking about the level of tin addition. As we will see later on, the present day emphasis is on the synergetic effect of tin and niobium in new zirconium alloys along with control of trace elements to derive the most optimum property in a cost effective manner.

To drive home the constraints of zircaloy production, I would like to mention here that the physical metallurgy of zirconium and that of titanium are very close - both belonging to group IV of the periodic table. Both of them exhibit an

allotropic transformation from the high temperature bcc (β) to the low temperature hcp (α) phase. The alloying behaviour of these two metals, however, is different because of the variation in their atomic size. As we are aware, the magnitude of the solute content that can be alloyed in a metal depends on the relative difference in the size of the solute atom (or the difference between the atomic size of the solvent metal and that of the solute should be as low as possible), in addition to other factors like the valence (should be of the same valence state as that of the metal), the electronegativity (preferably be the same) and the crystal structure (should be the same) . It is for the reason of the larger relative atomic size difference between the solutes and zirconium, that α-zirconium can accept solutes only to a limited extent. This has ultimately resulted in the development of only a handful of zirconium alloys for the nuclear industry (compared to α-titanium which can be alloyed to a large extent, resulting in a much wider choice of titanium alloys available to the aerospace industry).

In fact, as discussed earlier, only two series of zirconium alloys, viz., Zr-Sn and Zr-Nb have so far been developed, essentially due to restrictions imposed by the neutron absorption property on one side and the solubility limit of the alloying elements on the other. Important alloys of the Zr-Sn series are Zircaloy-2 (Zr-1.5 Sn – 0.1 Fe – 0.1 Cr – 0.05 Ni) and Zircaloy-4 (Zr – 1.5 Sn – 0.2 Fe – 0.1 Cr). Zircaloy-2 is used as the fuel clad material in PHWRs whereas zircaloy-4 is used as the fuel clad material in PWRs and PHWRs. Important alloys of the Zr-Nb series are Zr-1 Nb (fuel clad in VVERs), Zr – 2.5 Nb (pressure tubes in PHWRs) and Zr – 2.5 Nb – 0.5 Cu (garter springs in PHWRs).

Zirconium has many other special features, viz., (i) its anisotropy which invariably leads to the development of preferred crystalline structural orientations called "textures" during fabrication and the associated problem of irradiation growth of such textured alloys, (ii) its high reactivity with respect to oxygen, nitrogen and carbon which have also high solubility in the bcc (β) phase; and finally (iii) the low solubility of hydrogen in the hcp (α) phase, any excess hydrogen getting precipitated as hydride, leading to serious embrittlement.

Thermo-mechanical processing, therefore, assumes an important role in processing these alloys for meeting the stringent properties required for nuclear applications. The control of microstructure and texture by suitable thermo-mechanical processing is the key to the successful fabrication of nuclear structural components. In addition to chemical composition and microstructure, metallurgists have also to pay attention to the texture and dislocation density in zirconium alloys to be able to meet the specifications for applications in the nuclear industry. It may be noted that features such as dislocation density and crystallographic texture are rarely used as a part of the specifications of any engineering material in non-nuclear applications. Inspite of such stringent control of all these parameters that is needed in zirconium alloys, maintaining a high production yield is undoubtedly a very creditable achievement for NFC.

The problem of uniform corrosion at high temperatures has been well taken care of by alloy design and water chemistry control and this is no longer a life-limiting factor for the fuel clad tubes or the pressure tubes. However, the problem of localized attack due to fission gas on the inner surface of the cladding tube, resulting in stress corrosion cracking, is still a common failure mechanism and this problem has also been largely circumvented by graphite coating on the inner surface of the fuel tube to minimize both mechanical and chemical pellet-clad interactions.

In the recent past, the focus of R&D activities in zirconium metallurgy has been on several degradation mechanisms. The important degradation mechanisms of zirconium alloys can be grouped into two broad categories. These are (i) radiation induced mechanisms, namely,

irradiation embrittlement, irradiation creep and irradiation growth; and (ii) hydrogen induced mechanisms, namely, hydriding or hydrogen embrittlement, delayed hydride cracking and hydride blistering. All these limit the life of reactors.

Hydrogen absorption is about one-third in Zircaloy-4 as compared to Zircaloy-2. This is attributed to the absence of nickel in the former (maximum nickel content: 70 ppm as opposed to 300-800 ppm of nickel present in the latter). Similarly, in general Zr- Nb alloys absorb less hydrogen than Zr-Sn alloys which is attributed to the absence of intermetallics, viz., $Zr(Fe, Cr)_2$ and Zr_2 (Fe, Ni) in Zr-Nb alloys; these are normally present in Zr-Sn alloys.

As we are aware, hydrides in radial-axial orientation are the most detrimental and control of the orientation of hydride precipitates is important to reduce their detrimental effect on toughness reduction. In general, hydride orientation depends on crystallographic texture, tensile stress and microstructure. While it is the texture that primarily controls the hydride orientation, stress becomes a controlling factor when it exceeds a threshsold value. It may be noted that the threshold stress for hydride reorientation is higher for Zr-2.5% Nb alloy (180 – 220 MPa) than for zircaloy-2 (80-110 MPa) which, in turn, restricts the operating pressure of the coolant in respect of the use of these two alloys to 130 MPa and 95 MPa respectively.

Two incidents of Zr-2.5 Nb pressure tube failure in Canadian PHWRs have been attributed to delayed hydride cracking (DHC): (i) leakage of about 75 tubes near roll joint in Pickering 3 / 4 reactors during 1974-75; and (ii) catastrophic fracture of one tube in Bruce 2 reactor in 1986 during cold pressurization that was adopted to facilitate leak detection by acoustic emission testing. In Pickering-2 PHWR, a catastrophic failure of the Zircaloy-2 pressure tube had been caused by blister formation in 1983. Displacement of garter spring spacers led to pressure tube-calandria tube (PT-CT) contact and generation of a cold spot at the contact. This set up a thermal gradient, providing the driving force for hydrogen migration to the cold spot and initiation of hydride blister formation. Blisters cracked on reaching a critical size, leading to DHC and the cracks, on reaching a critical size, caused the catastrophic fracture of the Zircaloy-2 pressure tube.

NPCIL has already gone through a large scale pressure tube replacement programme which became necessary as the first generation Zircaloy-2 pressure tubes had already seen about eight full power years of service, beyond which the structural integrity of Zircaloy-2 pressure tubes cannot be assured for continued service. The decision for the removal of these pressure tubes from reactors was taken after systematic in-service inspection (ISI) of coolant channels. A semi- automatic remotised inspection system, known as BARCIS (BARC Channel Inspection System), for in-service inspection of coolant channels has been developed which utilizes three ultrasonic and three eddy current probes to measure wall thickness of pressure tubes, to detect flaws, if any, to locate the position of garter springs and to measure the gap between the pressure tube and calandria tube. A non-intrusive vibration testing technique has also been developed for shortlisting the probable contacting channels without the fuel load which are subsequently studied in detail by ISI to confirm the contacting of channels. Further, these in-service inspection techniques have been supplemented by several computer codes developed in-house, for the life management of PHWR coolant channels. The code 'SCAPCA' performs creep sag calculations to predict the gap between the calandria tube and pressure tube as a function of time. The computer code 'HYCON' performs calculation for hydrogen pick up in pressure tubes while the code 'BLIST' determines the time required to reach the critical blister size in a contacting channel based on the calculations of hydrogen pick up and its diffusion to a local cold spot, resulting in blister growth. BARC has also

developed the necessary SCRAPE tool for drawing samples from the inside surface of pressure tubes for establishing the hydrogen pick-up during service without affecting their service life.

Let me now draw your attention to some of the current programmes. It has recently been pointed out that the fracture toughness of the Zr-2.5 Nb pressure tube material is strongly influenced by trace amounts of chlorine and phosphorus present in it. The quadruple melting practice now adopted in NFC has already been able to achieve a level of chlorine below 0.5 ppm, and phosphorus less than 10 ppm, resulting in higher fracture toughness of this quadruple melted material. This quadruple melting practice has also reduced the hydrogen level in the as manufactured material to below 5 ppm. In this connection, it may be noted that as a thumb rule, one can take about one ppm hydrogen entry (in the Zr-2.5 Nb alloy) per full power year operation of a PHWR.

Coming to the calandria tubes, which experience much less severe service conditions, the most important concern is irradiation induced growth (at 80 °C) unlike irradiation creep for the coolant tube (under working pressure and temperature). We are aware that NFC has already established the fabrication technology for seamless calandria tubes by the pilgering route. Detailed characterization of seamless calandria tubes with respect to microstructure, texture and mechanical properties had already confirmed the suitability of this route for calandria tube production. However, data on the irradiation growth of this seamless material was not available in the early nineties when the first development of the pilgering route for calandria tubes was tried. Accordingly, a programme was initiated at BARC for the assessment of irradiation growth of samples taken from pilgered seamless tubes as well as from seam welded tubes. After generating the necessary radiation growth data and its assessment by safety authorities, NFC is now geared up to supply the required number of seamless calendria tubes for the 500 MW(e) reactor currently being built at Tarapur.

I would like to conclude my presentation by mentioning some of the future directions of our research activity in zirconium metallurgy. Today, we are considering increasing the burn up of our fuel for better economy in fuel cycle and there is an urgent need for the development of newer zirconium based cladding alloys which can survive along with the higher burn up fuel. I have indicated earlier that after years of research, it is now recognised that decreasing tin to a lower level (from the existing content of 1.2 – 1.7% to 1.2 %) in the specification range of Zircaloy-4 is beneficial. The synergy of low tin with added niobium as alloying elements in zirconium alloys has been shown to be beneficial not only for improved strength, ductility and corrosion resistance but also for the fabrication of such materials. In this context, mention must be made of the Zr-Sn-Nb series of alloys which have been developed; these include ZIRLO (Zr – 1.0 Sn – 1.0 Nb – 0.1 Fe), the 1-1-0.1 alloy of Westinghouse, which has the Russian equivalent of Alloy-635 for extended burn up fuel used in PWRs, and EXCEL (Zr – 3.5 Sn – 0.8 Nb – 0.8 Mo) for creep resistant pressure tubes in PHWRs.

In this connection, it is to be noted that Zircaloy-4 has a Total Circumferential Elongation (TCE) of about 15% initially which gets reduced to around 2% after 7000 MWD/Te of burn up. How to increase the initial TCE from 15% to about 22-25% is a major challenge to the metallurgists; such as increase will go a long way to extend the burn up of the fuel.

In short, our R&D efforts are for developing more "Fault Tolerant" or "Friendly Zirconium Alloys" which are more tolerant to flaws, more tolerant to abnormal environmental conditions, more resistant to irradiation damage and more immune to hydrogen induced degradation. The materials scientists and design engineers in BARC have always been working hand-in-hand with their counterparts at NFC for the process development

and optimization of zirconium alloy products. The present symposium is yet another example of the collaborative efforts between BARC and NFC in bringing together research scientists, design engineers, fabricators, along with reactor operating personnel and safety authorities on a common platform for discussing all aspects of zirconium technology, particularly to review the status of zirconium technology in the world and to provide the road maps for the future development work on various aspects of zirconium alloy production technology.

BIOLOGICAL INDICATORS OF ABSORBED RADIATION AND BIOLOGICAL DOSIMETRY

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Background

Biological indicators of radiation damage refer to measurable and quantifiable changes that take place in the biological system following exposure to ionizing radiations. Even though there are a large number of biological indicators, very few of them that show a consistent dose response relationship can be qualified as biological dosimeters. Cytogenetic changes in human peripheral blood lymphocytes and ESR spectroscopy of tooth enamel are the only techniques validated for biodosimetry. Biodosimetry constitutes an inseparable part of radiation protection programme and is being routinely used for investigation of suspected overexposure cases. In India, Biological dosimetry work was initiated in the year 1975 at Bhabha Atomic Research Centre (BARC). Chromosomal aberrations were scored in cultured peripheral blood lymphocytes. Since 1992, this work is being done by Radiation Biophysics group of the Radiological Physics and Advisory Division (RP&AD). Since the inception of biological dosimetry programme, six hundred and fifty eight suspected overexposure cases have been analysed by chromosomal aberration analysis (CAA). Micronucleus assay in cytochalasin blocked binucleated lymphocytes was also carried out in a few cases. Since our laboratory is the only place where CAA is undertaken for biological dosimetry, cases of suspected overexposure of radiation workers from industries (mainly industrial radiography), diagnostic radiology, research, and nuclear facilities are referred to us by the appropriate authorities. Health, Safety and Environment Group of the Bhabha Atomic Research Centre, coordinates this activity. Over 45,000 radiation workers from all over the country are covered by the Personnel Monitoring Services.Overexposure Investigation Committee (OEIC) appointed by the Chairman, Atomic Energy Regulatory Board (AERB), examines every case of overexposure and refers the same to the RP&AD for cytogenetic dosimetry. Nuclear Industry workers are referred through the Head, Health Physics Division. Health physics units also monitor the workers for internal exposures by area monitoring, bioassay and whole-body counting and other techniques. Cases referred are subjected to medical examination as well as to CAA. Reports of chromosomal aberration analysis are sent to the OEIC, AERB, for interpretation in the light of other investigations.

Biological Indicators of Radiation Damage

In most of the radiation accidents, physical dosimetric information is rarely available. Further, most of the accidental exposures are non-uniform involving either partial body or localized exposure to significant doses. In such situations,

Table 1 : Biological indicators of radiation damage for different exposure conditions

Whole body exposure	Partial-body/localized exposure	Accumulated chronic exposure
Manifestation of prodromal syndromes: Time course appearance and persistence of radiation sickness has importance in the triage of accident victims	Chromosomal aberration analysis (dicer coupled with dispersion analysis. Dose to the part of the body exposed can be assessed.	Translocation assay: Assay of stable chromosomal aberrations by fluorescence *in situ* hybridization technique with the help of chromosome specific probes.
Haematological indicators: a. Lymphocyte counts at 48-72 hours after exposure (100-500/mm^3 very serious: <100/mm^3 likely to be fatal) b.. Kinetics of fall of neutrophil counts: Earlier the fall in the number of neutrophils to 500/mm^3 poorer the prognosis	Skin damage indicators: a. transient erythema b. depilation c. fixed erythema (3-4 weeks) d. death of follicular cells e. reduction in hair width f. cytogenetic damage in follicular cells g. skin burns(large doses)	ESR dosimetry based on the analysis of stable CO_3^- radicals formed in tooth enamel has served as a biological dosimeter both for acute exposures in accidents as well as in cases of chronic accumulated exposures. Past exposures in A-bomb survivors could be assessed by ESR.
Cytogenetic analysis: chromosomal aberration analysis of dicentrics; micronucleus assay; premature chromosomal condensation to assess the number of breaks and translocation assay by chromosome painting	Reproductive system(male): a. Distribution of sperm cells at different stages of spermatogenesis by flow-cytometry (invasive) b. Fall in sperm counts at 60 days post-exposure. (oligospermia /aspermia)	Chromosomal aberration analysis with appropriate decay correction factors can provide a crude indication of absorbed dose. This method tends to underestimate the dose and shows a great deal of inter-individual variations.
Biochemical indicators: a. Creatin/Creatinine ratio in urine shows a dose dependent rise with radiation dose b. Excretion of amino acids such as β-amino isobutaric acid	Head exposure: Changes in the EEG patterns resulting from head irradiation. The response can be observed for doses above 0.25 Gy due to changes in the electrical activity of brain.	Mutation in the *Glycophorin A* locus results in a dose dependent modification of the RBC membrane proteins. This can be detected (among MN heterozygotes) by fluorescent antibody using flowcytometry.
ESR signals from tissues like tooth enamel, bone, materials carried by the individual such as sugar in pills and mother of pearl.	Bone marrow cell damage in samples derived from different parts of the body.	

physical dosimetry does not provide reliable dose estimate. It has now been realized that biological dosimetric techniques can play an important role in the assessment of absorbed dose. In recent years, a number of biological indicators of radiation have been identified. These include, the kinetics of onset and persistence of prodromal syndromes (radiation sickness) [1], cytogenetic changes in peripheral blood lymphocytes[2], hematological changes[3], biochemical indicators, ESR spectroscopy of biological samples [4], induction of gene mutations in red blood cells[5], cytogenetic and physiological changes in the skin[6] and neurophysiological changes[3]. The importance of accurate dose assessment in radiation accidents for the effective management of radiation accidents has been realised [7]. In general, the dosimetric information is derived by a combination of several different methods. Although some of these indicators may be purely qualitative or semi-quantitative, they have potential to serve as prognostic indicators. A summary of the different biological indicators and their specific usefulness under different exposure conditions is presented in Table 1. The role of cytogenetic techniques in peripheral blood lymphocytes (PBL) as biological indicators of

absorbed radiation is reviewed here. These include, chromosomal aberration analysis (CAA) for unstable aberrations (dicentrics+rings), micronucleus assay in cytokinesis blocked lymphocytes (MN assay), premature chromosome condensation (PCC) and translocation assay by fluorescence *in situ* hybridization (FISH).

Cytogenetic Techniques in Biodosimetry

Chromosomal aberration analysis

Chromosomal aberration analysis(CAA), based on the frequency of dicentric chromosomes in cultured peripheral blood lymphocytes, is so far the most reliable biological dosimetric technique[8]. The protocol for CAA is described in Fig 1. The physical appearance a normal metaphase is shown in Plate 1, and metaphases with dicentrics, tricentric, deletions and ring are shown in Plates 2 and 3. The background dicentric frequency in unexposed blood is very low and lies in the range of 5×10^{-4}–10^{-3}(1-2 in 2000 cells). The frequency increases with dose linear-quadratically for low LET radiations and linearly for neutrons and high LET radiations.

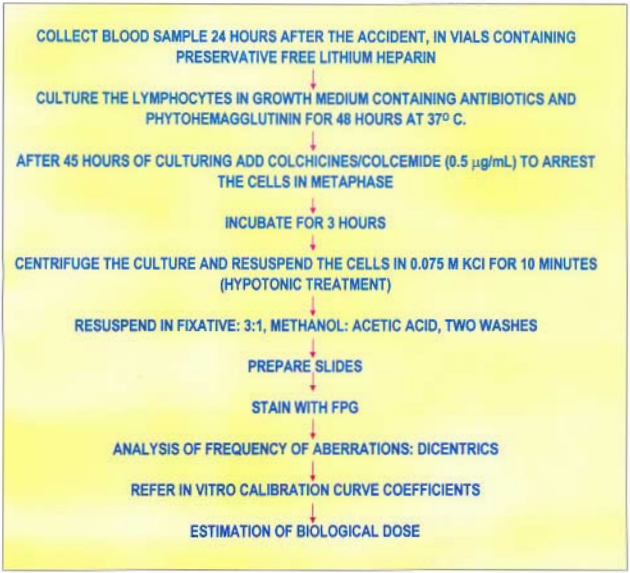

Fig.1 : Protocol for biological dosimetry by chromosome aberration analysis

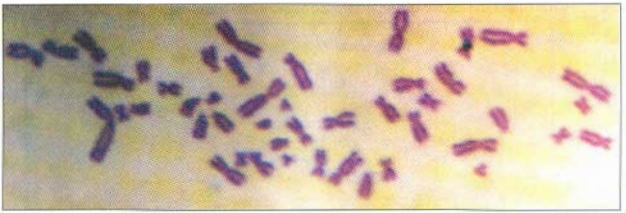

Plate 1 : Normal metaphase

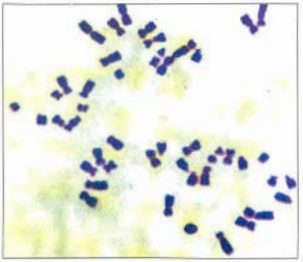

Plate 2 : Dicentrics, tricentrics and ring

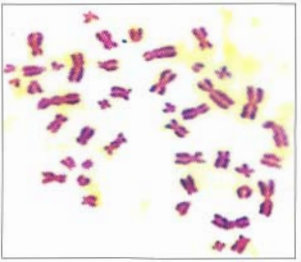

Plate 3 : Two dicentrics

The frequency of dicentrics "y" , varies with the dose "D" as follows:

$$y = c + \alpha D + \beta D^2$$

The linear (α) and quadratic (β) coefficients are characteristic of the type of radiation. For neutrons, the response is almost linear. Generally, 500 metaphases are scored per sample and the frequency of dicentrics, "y" is determined. Absorbed dose is assessed from the relationship,

$$D = \frac{\left[-\alpha + \sqrt{\alpha^2 + 4\beta y} \right]}{2\beta}$$

This dose refers to the equivalent whole body dose received acutely in an accidental exposure.

The lower limit for detection is 50 mGy of X-rays, 100 mGy of γ radiation and 10 mGy for fast fission neutrons. Different laboratories have obtained calibration curves for many different radiations. The value of calibration curve coefficients α and β are presented in Table 2. Estimates in the low dose region based on a few dicentrics are always associated with large Poisson errors. Mean dose estimates corresponding to 1-10 dicentrics scored along with 95% confidence interval are shown in Table 3. Uncertainties in the high dose region reduce significantly due to the large number of dicentrics scored. The mean dose estimates corresponding to dicentric frequencies in the range of 0-2, derived by scoring 500 metaphases and the upper and lower 95% confidence intervals are described in Fig.2.

One of the main advantages of CAA is its radiation specificity. Only a very few clastogens

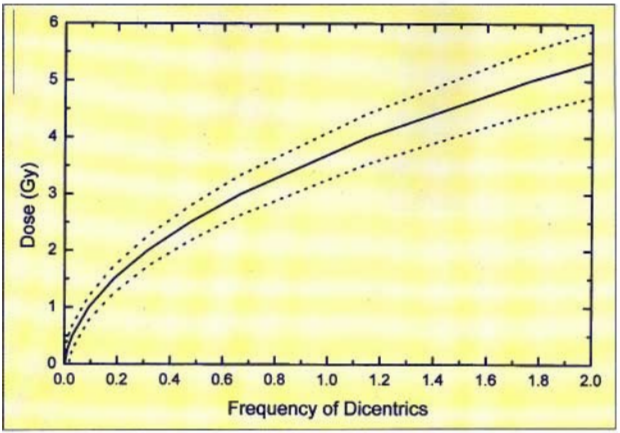

Fig.2 : Mean dose estimate and 95% confidence interval corresponding to different dicentric frequencies for 500 metaphases scored

Table 2 : Calibration curve parameters for different radiations

Type of radiation	Dose rate (Gy/min)	$\alpha \times 10^{-2} \pm$ S.D. Gy^{-1}	$\beta \times 10^{-2} \pm$ S.D. Gy^{-2}
^{60}Co gamma rays	3 mGy/min	1.8 ±0.8	2.9 ±0.5
^{60}Co gamma rays	0.5 Gy /min	2.7±0.5	6.5±0.3
HTO β rays	0.1-1.5 Gy/min	8.23±0.4	6.4 ± 0.2
250 keV X-rays	0.5 Gy/min	3.8±0.7	6.8±0.3
15MeV electron	1 Gy/min	0.6±0.1	5.7±0.6
Fission neutrons (1MeV)	20 mGy/min	95	-
Fission neutrons (0.4 MeV)	2-70mGy/min	90±7	
^{252}Cf neutrons	2.5mGy/min	60±2	
Fission neutrons 0.7MeV)	0.5Gy/min	83±1	
π⁻ mesons (peak)	20 mGy/min	23 ± 2	4.8 ± 0.9

Table 3 : Dose estimates based on dicentric chromosome analysis in low dose region

Number of dicentrics in 500 metaphases	Dose estimates with lower and upper confidence limits (95%) for 500 metaphases scored (mGy)		
	Lower	Mean	Upper
0	0	17	100
1	0	65	160
2	0	120	225
3	25	160	270
4	65	200	310
5	90	235	340
6	130	270	375
7	160	300	405
8	190	330	435
9	220	358	465
10	250	385	490

such as bleomycin, endoxan etc. can be confounding factors for this assay. Doses over a wide range of 0.1 -6 Gy can be detected by this technique. CAA has provided very reliable dose estimate during the Chernobyl [9] and Goiania [10] accidents. Following acute radiation exposure, dicentric frequency remains unaltered for several weeks, but decays to half the value in around 180 days [11]. Subsequent reduction in the signal occurs more slowly with an approximate half-life of 3 years, as seen in the case of low dose and chronic exposures. Hence, doses from past exposures can be assessed with reasonable reliability by applying appropriate correction factors. Some individual differences in the response for the induction of CA may arise due to variations in genetic composition and DNA repair capacity. Hence, CAA can serve as a useful indicator of biological response of a specific individual. One of the most important limitations of this technique is the requirement of a well-experienced scorer. Further, the scoring of dicentrics is not only time consuming, but also not easily amenable to automation due the complexity of the image of dicentrics to signal processing systems. Recent advances such as metaphase scanners and centromeric painting by antikinetocore antibodies can enhance the scoring speed and reduce errors in the identification of dicentrics. In accidents involving exposures in the lethal range (3-6 Gy), suffice it to score 25-50 metaphases for providing the preliminary information required for medical management of victims. However, at higher doses most of the severely damaged cells fail to go through the division and it may be very difficult to find even a few metaphases.

CAA in non-uniform exposures : In uniform whole body exposures, the radiation induced dicentric chromosomes follow a perfect Poisson distribution. However, in most accidents the exposures are non-uniform, resulting in a fraction of lymphocytes receiving a much larger dose as compared to those in the unexposed part of the body. As a result, the total aberrations scored are concentrated in a few damaged cells, which reach the metaphase. The degree of overdispersion can be assessed by the dispersion coefficient u [12]

$$u = \frac{(N-1)\,\sigma^2/y - (N-1)}{[2\,(N-1)\,(1-1/Ny)]^{1/2}}$$

where, N is the total number of cells scored; y the frequency of dicentrics and σ^2 its variance. A positive value of u indicates overdispersion, a negative value an underdispersion and a zero

value a perfect Poisson distribution. In accident situations, a value greater than 1.96 suggests a partial body or localized exposure. The mean dicentric yield corresponding to the fraction of lymphocytes exposed can be derived by the relationship, $Y = [1- e^{-Y}]\ n\ /\ (N-N_0)$, where n corresponds to the number of dicentrics in N cells scored; and N_0, the number of cells free of any dicentrics. The dose to the exposed fraction of lymphocytes can be derived from the mean yield Y, by referring to an appropriate calibration curve. Since the dicentric frequency obtained is a product of the mean yield Y and the fraction of the lymphocytes exposed (f), the latter can be easily calculated. We have validated this method in an actual radiation accident involving a partial body exposure to ^{192}Ir industrial radiography source [11]. Dicentric yield was only 0.20 but the dispersion indexes u varied between 8.7-15.3 during the first 3 months after exposure, thus suggesting a very non-uniform exposure. This was evident from the severe radiation burns seen on the thighs and hips of the victim. Mean yield Y of 1.65 obtained in this case corresponds to an average dose of 4.5 Gy to approximately 50 % of the body volume. The results obtained here were in agreement with physical dosimetry by reconstruction method. Even in the case of Goiania accident, some victims showed significant overdispersion, whereas many others with known non-uniform exposures did not [10]. This discrepancy may be due to the protracted nature of irradiation from internally deposited ^{137}Cs and external contamination. Hence, dispersion analysis can be reliably used only in acute partial body external irradiation.

Biological dosimetry for mixed neutron and gamma exposures (criticality accidents) [8]: In criticality accidents, persons are exposed to mixed fields of neutrons and gamma rays. In such situations, it is easy to know the n: γ ratio by physical methods. The neutron and gamma doses are assessed as follows:

a. Culture the blood samples and obtain the frequency of dicentrics (y)
b. Assume that the entire yield of dicentrics is due to neutrons and calculate the neutron dose (D_n) by referring to the calibration curve for neutrons.
c. With the knowledge of n: γ ratio, calculate the gamma dose ($D\gamma$).
d. Calculate the dicentric yield corresponding to $D\gamma$ from the gamma calibration curve.
e. Subtract this yield from the dicentric frequency (y) to determine the neutron contribution.
f. From the result of step 5, calculate the neutron dose.
g. Once again refer to the n: γ ratio as in step 3 and recalculate the gamma dose.
h. Repeat the steps until consistent estimates of doses are obtained.

Premature chromosome condensation (PCC)[13]

Peripheral blood lymphocytes in G_0 phase can be successfully fused with cultured CHO cells in mitotic phase using polyethylene glycol. This leads to premature condensation of the chromosomes in interphase lymphocytes. The 46 chromosomes appear with single chromatids. At this stage, damage induced by radiation appears as excess breaks and can be correlated with the dose. An important advantage of this method is that the information on exposure can be derived within 3-4 hours of obtaining the blood sample. Further, since the technique does not involve cell division, the artifacts associated with post-irradiation stimulation and progression through the cell cycle does not interfere with the analysis. Even in accidents involving cells exposed to doses in excess of 5 Gy, the cells can easily undergo condensation although they may fail to reach metaphase. PCC technique should be specifically useful in such situations. In spite of the speed of analysis, this technique is in use in very few centres.

Micronucleus assay (MN assay)

Micronuclei refer to small nuclei formed from chromatin material, which lag behind and does not get included in either of the daughter nuclei

formed during cell division. Exposure to radiation and other clastogenic agents results in a dose dependent increase in the frequency of MN.

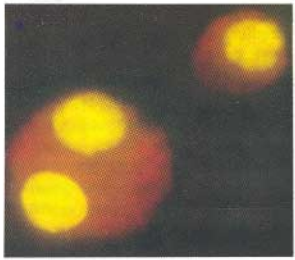

Plate 4 : Normal binucleated cell and mononucleated cell

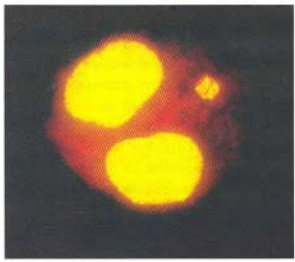

Plate 5 : Binucleated cell with one micronuclei

Dividing cells are blocked at cytokinesis using cytochalasin B, in order to visualize the induced MN. A few laboratories have explored the possibility of scoring MN as a method for biological dosimeter [4, 15, 16]. One of the important advantages with this technique is the simplicity and the speed of scoring. A technician can be trained to score these events within a very short time and scoring does not require highly skilled and experienced personnel as in the case of dicentric analysis. The binucleated cells with and without MN in acridine orange stained preparations are shown in Plates 4 and 5 respectively. Several thousand binucleated cells can be scored in a person-day as compared to 100-150 metaphases per day for dicentric analysis. Further, the possibility of automation for MN assay in the near future appears very bright. We have recently addressed to the problem of background MN frequency variation[16].

Among the 25 samples analysed, the background frequency varied from 6×10^{-3}-30×10^{-3} per binucleated cell; the mean value was 12 ± 4 per 1000 cells [16]. Dose response curve for the induction of MN derived by *in vitro* irradiation of blood samples from healthy donors, suggest some degree of variation as compared to those reported by others. In general, the calibration error for MN assay appears to be much larger as compared to that for CAA. Furthermore, our results show a significant deviation from Poisson distribution (over dispersion) for MN. This reduces the possibility of using this assay for distinguishing partial body exposures from uniform whole body exposures. Even though there are claims that MN assay can detect absorbed doses of the order of 50 mGy, large individual variations in background frequency do not permit reliable estimates of doses less than 0.25 Gy. In accident situations involving absorbed doses in the range of 2-6 Gy, very reliable information can be obtained within 3 days after the exposure. In accidents involving exposure to a large number of people, MN assay may be particularly useful, when well-trained technicians are not available to score dicentrics. The prospects of automation are much better for MN assay as compared to CAA.

Fluorescence in situ hybridization assay

Radiation induced unstable chromosomal exchanges like the dicentrics, rings and deletions decay with $T_{1/2}$ of 1-3 years depending upon the exposure condition. As a result, there is a considerable uncertainty in this dosimetry for past exposures [8,11]. Scoring stable chromosomal exchanges such as translocations is a possible approach to overcome this problem. Conventionally banding technique, which is very

tedious and time-consuming, scores translocations. Even for automation, the quality of the banding preparations should be very good. Recently the availability of whole chromosome specific libraries has enabled painting of individual chromosomes by fluorescence *in situ* hybridization technique. Translocations involving exchange of parts between the painted chromosomes and counter-stained (unpainted) chromosomes are visualised as bicoloured structures. FISH assay not only makes the identification of translocations very easy, but also increases the sensitivity by its ability to score events, which the conventional banding may fail to detect. In recent years, many laboratories have explored the potential of FISH assay of translocations as a biological dosimeter [17-21]. Since only a part of the genome is painted (10-20%), the information for the whole genome is derived by extrapolation of the response obtained for the painted fraction. Since it is likely that individual chromosomes may differ in their radiosensitivity, there is a need to obtain calibration curves with different cocktails of painted chromosomes.

Some of the previous reports suggest the usefulness of FISH assay for retrospective biological dosimetry of radiation [22, 23]. Even though a good agreement was seen for survivors of A-bomb, doses were underestimated for Y 12 accident victims [24]. It must be emphasised that more information from a large number of chronic exposure cases is necessary for validation of this technique as a biodosimeter.

Other Methods for the Assessment of Biological Damage

Neutron activation analysis

Neutron activation results in the formation of ^{24}Na and ^{32}P. These isotopes can serve as useful indicators of neutron dose [25]. Measurements of activity of ^{24}Na with HPGe detector can detect neutron doses of the order of 10mGy. The activity can be measured in a number of biological samples such as blood, urine, hair, or fingernails. The ratio of the measured activity of ^{24}Na in blood and ^{32}P in hair can provide useful information on the neutron spectra. Activation of extraneous objects such as clothing, coins, jewellery, wrist watch and other metallic objects carried by the individual will also be useful in dose assessment.

Electron spin resonance studies

ESR studies on biological samples such as bone, teeth, hair, nails and skin following irradiation can detect doses as low as 0.3Gy- several Gy [4]. The intensity of ESR signals is greater for photons of lower energy and weaker for neutrons. Lethal as well as sublethal doses can be detected by this technique. The signals in the tooth enamel are very stable and can be detected even after a lapse of 2-3 decades. This technique has provided dose estimates consistent with chromosomal aberration analysis in the case of Chernobyl accident victims [26]. Bone samples from the amputated leg of a heavily irradiated individual at San Salvador, analysed by ESR showed good correlation with the physical dose estimates [27]. At present, this technique holds promise for quick estimation of dose. Further, the ESR technique is the only reliable method for retrospective biological dosimetry. This has been validated by the dosimetry carried out on the A-bomb survivors. The development of portable ESR spectrometer and microwave cavities have enabled *in vivo* dose assessment without the extraction of the tooth [28].

Sperm analysis

Distribution of sperm cells in the spermatogenic cycle is disturbed as a result of radiation exposure. This can be detected and quantitated by flow-cytometry of germ cells from the testes. This method is useful only in the case of males. In addition to this, the sperm count itself provides an indication of the dose to the testis. Generally, the sperm counts never decrease up to 40 days due to the radioresistance of germ cells in the later part of the spermatogenic cycle. A steep fall

occurs in around 4 months. This method was successfully used to detect the dose to the male reproductive system among the Marshelese islanders exposed to the fallout from thermonuclear device [29].

Neurophysiological dosimetry

Irradiation causes changes in the concentration of many chemical mediators associated with central nervous system function. These include acetylcholine, cholinesterase balance, aspartic acid, adrenergic amines, gamma amino butyric acid, membrane permeability and acid base balance. These could result in changes in cerebral electrical activity. The changes appear immediately after irradiation and remain stable for quite long. These changes result in the altered patterns in electroencephalograph (EEG). The response can be observed even for doses greater than 0.25 Gy. The method is fairly simple, non-invasive and very useful where CAA cannot be carried out. The changes persist even after long duration [30].

Dosimetry using skin

Visible changes in skin colour, erythema, desquamation, ulceration, necrosis and depilation are good indicators of damage. Chromosomal damage in cells from plucked hair may be useful to study the dose distribution in non-uniform exposures. Damage to follicular cells is an invasive procedure and there is limited human experience. Reduction in the hair width caused by damage to hair follicles can be observed only after 2-3 weeks. More work is necessary to validate this method in human beings [31, 32].

Objectives of biological dosimetry

- **To confirm or reject the findings of physical dosemeters and to distinguish between genuine and non-genuine exposures.**
- **To detect a suspected exposure when there is no information from physical dosemeters, such as in the case of a person who is not routinely monitored or a radiation worker not wearing a badge.**
- **To provide reassurance to those who are false positive on physical dosemeters which got exposed while being not worn, or maliciously irradiated.**
- **To detect the average dose to the body in the case of very non-uniform exposures. In such situations the dosemeters may indicate a very high or low dose depending upon the irradiation geometry.**
- **To distinguish between protracted and acute exposures in accidents. CAA provides a purely biological response taking into consideration the duration of exposure and accounting for the possible repair of radiation damage.**
- **To detect the dose to the exposed part of the body and the fraction of the body exposed, in accidents involving inhomogeneous exposure(partial-body, or localised). Dispersion analysis of the chromosome aberration data indicates the deviation from a Poisson distribution, which in turn is a measure of the non-uniformity of exposure.**
- **To assist the triage of victims in accidents involving large number of exposed individuals. In the medical management of radiation accidents CAA can serve as a reliable prognostic indicator.**

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"ZIRCONIUM-2002" SYMPOSIUM

A symposium "ZIRCONIUM – 2002" was organised at BARC during September 11-13, 2002, under the auspices of Board of Research in Nuclear Sciences (BRNS), Department of Atomic Energy, Government of India. This symposium provided a forum for the exchange of state-of-the-art knowledge and expertise on various aspects of zirconium metallurgy as related to its use in the nuclear power industry. Over 300 professionals from all over the country, who are engaged in research, production, fabrication and evaluation activities related to zirconium components, participated in the symposium. Distinguished experts from USA and South Korea also shared their experiences with the participants. The topics covered in the symposium included: processing from minerals to metal, alloy development and fabrication, deformation, texture and microstructure–property

Dr Anil Kakodkar, Chairman AEC, delivering the inaugural address at the symposium " Zirconium – 2002 ". Others on the dais are (from left to right) Dr P. K. De, Head, Materials Science Division & Convener, Mr B. Bhattacharjee, Director, BARC, Dr C. Ganguly, Chief Executive, NFC and Dr S. Banerjee, Director, Materials Group, BARC.

correlation, corrosion and pellet-clad interaction, hydrogen related problems, in-service inspection, post-irradiation examination and quality assurance. Nine talks in plenary sessions were delivered by eminent speakers. More than a hundred technical papers were presented by experts on different aspects of zirconium technology. The pre-prints of all these papers have been brought out in a volume edited by Dr. P. K. De.

The symposium was inaugurated by Dr Anil Kakodkar, Chairman, Atomic Energy Commission, and Secretary, Department of Atomic Energy. In his inaugural address, Dr Kakodkar outlined the success story of indigenous development of zirconium technology and pointed out some of the future directions of research in this area. He stressed the need for the development of defect tolerant zirconium alloys for cladding and pressure tube applications and for research on technologies for bonding and joining of zirconium alloys. He also pointed out that the removal of zirconium isotopes with high neutron absorption cross sections can lead to a substantial neutron economy. The inaugural function was presided over by Mr B. Bhattacharjee, Director, BARC. Dr Srikumar Banerjee, Director, Materials Group, BARC, welcomed the delegates and Dr C. Ganguly, Chairman, Organising Committee and Chief Executive, NFC, in his talk narrated how DAE scientists met the challenging task of in-house development of the total technology of zirconium. Dr P. K. De, Head, Materials Science Division, BARC, and convener of the symposium, presented the vote of thanks.

TRAINING SCHOOL GRADUATION FUNCTION OCES45 & OCEP11

The Graduation Function of OCES45 (Orientation Course for Engineering Graduates & Science Post Graduates) and OCEP11 (Orientation Course for Engineering Post Graduates) was held on August 29, 2002 at the Central Complex Auditorium, BARC.

Ms G.S. Saritha of Electronics Engineering discipline who stood first among all 12 OCES45 disciplines receiving the Homi Bhabha Prize from the Chief Guest Mr Suresh P. Prabhu, Hon. Member of Parliament

Mr Suresh P. Prabhu, Honorable Member of Parliament and former Cabinet Minister for Power, gave away the Homi Bhabha Prizes and delivered the Chief Guest's address.

Dr Anil Kakodkar, Chairman, AEC and Secretary, DAE, and Mr B. Bhattacharjee, Director, BARC, presided over the function.

120 OCES45 and 23 OCEP11 Trainee Scientific Officers' graduated and were inducted into the DAE family. The following is the list of Homi Bhabha Prize Awardees.

Sr. No.	Discipline	Name
1.	Mechanical	Mr Abhishek Basak
2.	Chemical	Mr Anil Gordhanbhai Patel
3.	Metallurgy	Mr Kamlesh Chandra
4.	Electrical	Mr Kalyan Chakaravarthy Madala
5.	Instrumentation	Mr Dipak Dwarkadas Patel
6.	Electronics	Ms Saritha G.S.
7.	Computer Science	Mr Amar Deep Kumar
8.	Civil	Mr Pavan Kumar Emani
9.	Physics	Mr Himanshu Kumar Poswal
10.	Chemistry	Mr Madhava B. Mallia
11.	Bioscience	Mr Manish Goswami
12.	Environmental	Ms Kothai G.
13.	M.Tech. (Mechanical)	Mr Jagannath Mishra

भाभा परमाणु अनुसंधान केंद्र के वैज्ञानिकों को सम्मान

BARC SCIENTISTS HONOURED

• डॉ ए.एस.प्रधान, अध्यक्ष, अंशांकन एवं मात्रा अभिलेखन अनुभाग, विकिरणकीय भौतिकी एवं सलाहकार प्रभाग, भापअ केंद्र के "विकिरण से उद्भासन मात्रा" (Doses from Radiation Exposures) पर वर्ष २००१-२००५ तक की अवधि के लिए अंतराष्ट्रीय विकिरण संरक्षा आयोग (ICRP) समिति - २ का सदस्य बनाया गया है । उन्हें यह गौरव विकिरण संरक्षण, विशेषतः तापसंदीप्तिशील मात्रा मिती (TLD) कार्मिक मानीटरन एवं विकिरण मात्रा मिती के क्षेत्र में उत्कृष्ट योगदान के लिए प्रदान किया गया । आइसीआरपी एक स्वैच्छिक अंतरराष्ट्रीय निकाय है । इसके द्वारा विकिरण संरक्षण के लिए सिफारिशें दी जाती है जिन्हें लगभग सभी देशों द्वारा अंगीकृत किया जाता है । वर्ष १९८१ से डॉ. प्रधान संयुक्त गणराज्य (UK) द्वारा प्रकाशित "रेडियेशन प्रोटेक्शन डोसिमेट्री" नामक अंतरराष्ट्रीय पत्रिका के संपादकीय बोर्ड के सदस्य हैं । डॉ प्रधान जर्मनी के अॅलेक्सैंडर वॉन हमबोल्ट फाउंडेशन के भूतपुर्व अध्येता रह चुके है और १७० से अधिक शोध पत्र प्रकाशित कर चुके हैं । वे दक्षिण पूर्व एशिया में विकिरण संरक्षा के समन्वयन के क्षेत्र में क्षेत्रीय सहकारी समझौता/अंतरराष्ट्रीय परमाणु ऊर्जा अभिकरण (आरसीए /आईएईए) के राष्ट्रीय परियोजना समन्वयक भी हैं । वे मुंबई विश्वविद्यालय के भौतिकी विषय के लिए अनुसंधान गाइड भी हैं ।

Dr A. S. Pradhan, Head, Calibration & Dose Records Section of Radiological Physics and Advisory Division, BARC, has been nominated a member of Committee-2 of the International Commission on Radiological Protection (ICRP) on "Doses from Radiation Exposures" for the period 2001-2005 for his outstanding contribution in the field of radiation protection, especially thermolumine-scence dosimetry (TLD), personnel monitoring and radiation dosimetry. ICRP is an independent international body, which offers its recommendations for radiological protection that are adopted by almost all countries. Since 1981, Dr Pradhan has also been on the Editorial Board of an international journal entitled "Radiation Protection Dosimetry" published from UK. Dr Pradhan, a former fellow of Alexander von Humboldt Foundation of Germany, has published more than 170 research papers. He is also the National Project coordinator of RCA/IAEA (Regional Cooperative Agreement/International

Atomic Energy Agency) in the area of armonization of radiation protection activities in South East Asia. He is also a research guide in physics for the University of Mumbai."

• डॉ. एस. के. एच. औलक, अध्यक्ष, प्लासमा प्रयोगशाला, उच्च दाब भौतिकी प्रभाग, भापअ केंद्र, को सघन चुंबकीकृत प्लासमा पर अंतरराष्ट्रीय वैज्ञानिक समिति (ISC-DMP) के भारतीय प्रतिनिधि के रूप में नामित किया गया है। यह निकाय अंतरराष्ट्रीय सघन चुंबकीकृत प्लासमा केंद्र (ICDMP), वॉरसा, पोलैंड के कार्यों को देखता है। इस अंतरराष्ट्रीय केंद्र को दिनांक २४ अगस्त, १९९९ को यूनेस्को और पोलैंड के राष्ट्रीय परमाणु ऊर्जा अभिकरण के बीच समझौते के अंतर्गत इन्स्टिट्यूट ऑफ प्लासमा फिजिक्स एंड लेसर माइक्रोफ्यूजन में स्थापित किया गया है। सघन चुंबकीकृत प्लासमा पर अंतरराष्ट्रीय वैज्ञानिक समिति मे निम्नलिखित देशों के प्रतिनिधि है: फ्रांस, अर्जेंटीना, संयुक्त गणराज्य, चिलि, रूस, जापान, ज्रेक गणराज्य, सिंगापुर, चीन इटली, पोलैंड, जर्मनी, यूक्रेन, अमरीका, मलेशिया,रूमानियाऔर बलगारिया। इस केंद्र के मुख्य कार्य है :

- अनुसंधान,प्रशिक्षण एवं शिक्षा के माध्यम से सघन चुंबकीकृत प्लासमा भौतिकी तथा संबंधित प्रौद्योगिकी का ज्ञान बढाने में योगदान;
- अनुसंधान शोधों के व्यावहारिक अनुप्रयोग करने को बढ़ावा देना और कार्यान्वित करना
- अंतरराष्ट्रीय वैज्ञानिक सहयोग, विनिमय एवं संचार का संरक्षण
- वैज्ञानिक उपलब्धियों पर सूचना का प्रसार

इससे सघन चुंबकीकृत प्लासमा के क्षेत्र में कार्यरत भारतीय वैज्ञानिकों और अंतरराष्ट्रीय वैज्ञानिक समुदाय के बीच गहरे संबंध का आधार बन सकता है।

Dr S.K.H. Auluck, Head, Plasma Laboratory, High Pressure Physics Division, BARC, has been nominated as the official representative of India on the International Scientific Committee on Dense Magnetized Plasma (ISC-DMP), a body that oversees the activities of the International Center for Dense Magnetized Plasmas (ICDMP), Warsaw, Poland. This International Center has been set up under an agreement dated August 24, 1999 between UNESCO and the Polish National Atomic Energy Agency at the Institute of Plasma Physics and Laser Microfusion. The International Scientific Committee for Dense Magnetized Plasmas has representatives of the following countries : France, Argentina, UK, Chile, Russia, Japan, Czech Republic, Singapore, China, Italy, Poland, Germany, Ukraine, USA, Malaysia, Romania and Bulgaria. The main tasks of the Centre are :

- contribution - through research, training and education - to the progress of knowledge in dense magnetized plasma physics as well as related technology ;
- promotion and implementation of research findings into practical applications;
- support and maintenance of international scientific co-operation, exchange and communication;
- dissemination of information on scientific achievements.

This is expected to lay a firm basis for closer interaction between Indian Scientists working in the field of Dense Magnetized Plasmas and the International Scientific Community.

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