MATERIAL RESEARCH WITH BEAMS AT CYCLOTRON ENERGIES

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ABSTRACT

The use of cyclotron beams for material research covers a wide range from analytical research to radiation damage, materials modification and to solid state physics. The application of particle beams in these different areas will be described, and examples for these applications will be given. Up to now very few facilities are equipped to fully supply all the needs material scientists have. The requirements for cyclotron facilities catering to material science, solid state physics and ion beam applications are given.

1. INTRODUCTION

The use of particle beams in material research is on a steady increase, as the response to a survey at cyclotron facilities indicates. This type of research can be classified into the following areas:

-Analysis, used to determine material composition, impurities and depth-distributions of either unwanted impurities or of atoms, which have been introduced especially to change the properties of the original material.

-Investigation of radiation damage to materials, with the aim to understand the mechanism of damage and thus hopefully provide a means to design materials with better radiation resistance.

-Modification of materials by use of particle beams to find ways to create materials with new properties which could not be obtained by any other way, or to improve on already existing properties of the material under investigation.

-Investigations in solid state physics providing information on internal properties of solids, or on internal defects and helping us to understand the solid state of materials better. This type of research is often referred to as "nuclear solid state physics".[1)]

So far only a few facilities exist, which provide the necessary equipment for some of the investigations discussed. Generally the material research experiments are being done as a side line at facilities which have been built mainly for the use in nuclear physics, and we cyclotron builders need to become aware of the different requirements for the experimental equipment as well as for beam properties needed for material research. The only exception is Japan,[2)] where a facility is being built solely for the use in material research, with all the required ancillary equipment, to perform the full width of investigations described.

In the following I want to describe the different types of research with particle beams in material science in more detail, and give examples of work which is going on at different cyclotrons laboratories.

2. MATERIAL ANALYSIS WITH PARTICLE BEAMS

To analyze materials, and to determine amount and distribution of impurities, several different methods are being applied. A short description of these methods and of their applications will be given.

2.1 Charged Particle Activation Analysis (CPAA)

The method of charged particle activation analysis is based on the use of nuclear reactions to determine the presence of certain atoms in a material. It is therefore also often referred to as "nuclear reaction analysis" (NRA). This method is used primarily to determine low concentrations of impurities. This is especially important for the development of semiconductor devices, where the type and amount of impurities need to be monitored and controlled to concentrations down as low as possible. Other applications are metallurgy, biology and medicine, archeology and environmental technology. In general CPAA is rather complex and therefore not useful for routine applications. Its high sensitivity however makes this method very important for the development of procedures in the manufacturing of high purity materials. The sensitivity of this method reaches down to concentrations as low as 1 to 10 ng/g. A relatively recent account of this method has been given by Strijckmans et.al.[3)] The group in FZR Rossendorf near Dresden have used this method also and reached sensitivities as low as given above.[4)]

2.1.1. Thin layer activation (TLA)

A special case of CPAA is the method of thin layer activation. It was first developed by Schatz et. al.[5] and is now used as a research tool to help in the design of automotive parts and machine tools and to measure erosion and corrosion on pipes etc.. The method uses nuclear reactions of light and medium heavy ions to produce trace quantities of radioisotopes with well defined depth distributions near the surface of the parts, which one wants to monitor or improve. This way for instance the wear on motor parts can be observed in actual operating conditions by simply monitoring the activity in the motor oil with a simple radiation detector. This method is now in use in the automobile industry in Germany, the United States, Great Britain and Japan.[6] Also at VEC in Calcutta TLA - studies for motor parts have been performed.[7]
Originally the method of TLA was mainly used for metallic surfaces, where the particle beam could be directly applied to the surfaces to be studied. For nonmetallic, insulating or composite materials no nuclear reactions are available to use the direct beam on the material. In these applications the use of ^{7}Be has become the probe of choice, and developments at several laboratories are under way to produce ^{7}Be via the ^{7}Li(p,n)^{7}Be reaction and simultaneously recoil implant the probe nucleus into the sample for TLA investigations. At NSCL in East Lansing[8)9)] the production of ^{7}Be via a spallation process and successive acceleration has been successfully demonstrated. KFK Karlsruhe[10)], in a cooperation with the Kurchatov Institute, is using the reaction mentioned above to produce the ^{7}Be and recoil implant it into the sample. These two institutions also are developing a Be ion source with the aim to produce eventually an accelerated beam of ^{7}Be ions.

2.2 Neutron Radiography (NR)

Neutron radiography[11)] has long been a method of choice in material research, often applied in investigations of paintings etc..
In the past mostly reactors have been used. However there is now a proposal in Japan[12)] to produce neutrons for radiography with an intense cyclotron beam.

2.3 Rutherford Back Scattering (RBS)

This is probably the oldest ion beam method. It is based on the elastic back scattering of ions from the surface or the bulk of a material and it was used around 1910 by Rutherford in the first investigation of atomic structure. This method gives information about the different atoms in the material hit by the beam simultaneously, but often the peaks overlap with a continuum, due to signals from the bulk and the energy loss of the scattered particle in the material. This leads to an ambiguity which presents the main limitation to the use of this technique. Nevertheless this method is very useful in routine applications, especially when samples are compared relatively to each other. The typical sensitivity of RBS is about 0.1 atomic % with a depth resolution of 10 nm at a penetration of about 1 μm.[13)]

2.4 Elastic Recoil Detection Analysis (ERDA)

The ERDA-method is closely related to the RBS-method. Here one does look at the elastic recoil atoms coming from the sample instead of the beam particles scattered from the sample as in RBS. Due to the kinematics one preferably uses a heavy ion beam for ERDA, looking at all the lighter recoils coming from the sample, because the energy transfer to the recoiling ion is optimized this way. With ERDA one can measure simultaneously several different ions and their depth profiles, provided one determines the mass and energy of all the recoiling ions separately. In order to avoid nuclear reactions in the sample one tries to stay with the beam energy below the Coulomb barrier. This way the sample will not be activated at all and it can be handled after the ERDA analysis immediately. At VICKSI a high energy ERDA facility is being installed, using an ^{129}I beam of about 360 MeV. In Fig. 1. a sketch of the experimental setup

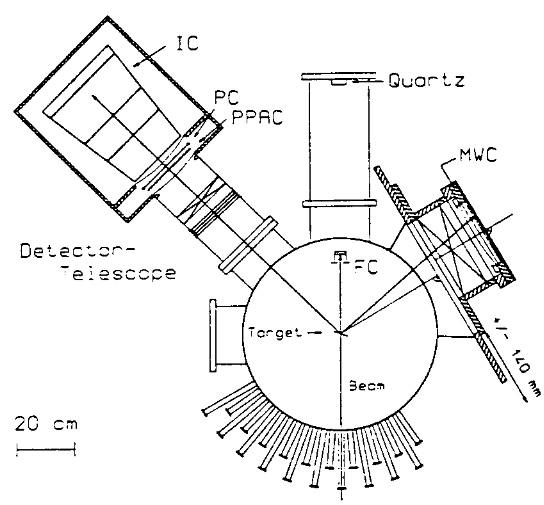

Figure 1 Sketch of the experimental ERDA setup. The scattering chamber is mounted on a rotatable support, with two large-area detectors attached.

is depicted. The detector telescope consists of a parallel plate avallange counter (PPAC), a proportional counter (PC) and an ionisation chamber (IC).The second detector is a low-pressure multiwire chamber (MWC). Tests have shown the great power of this method especially when combined with detectors and detection methods used for nuclear physics.[14)]

At the TU Eindhoven Rijken et. al.[15] using high energy α-particles, coincidence and pulse shape discrimination have also developed this method to obtain background free spectra and a depth resolution of 5 nm. In Brazil da Silva et al.[16] have recently used ERDA for Hydrogen profiling in surfaces of metallic materials.

2.5 Particle Induced X-ray Emission (PIXE)

The PIXE-method is mostly used with a relatively low energy proton beam. Often this beam is transferred out of the vacuum of the accelerator and furthermore used in conjunction with a microbeam. The method is based on the use of a beam particle to kick an electron out of the inner shell of a sample atom, and then to look at the characteristic X-rays coming from the sample with an appropriate detector. Practically all elements can be detected this way simultaneously. The absorption of the characteristic X-ray in the material surrounding the detector determines the limit for the lighter elements, so that in general elements lighter than sodium (Z = 11) can not be detected this way. The PIXE-method is in wide use at many places for all kinds of measurements pertaining to environment, health, biology, archeology and others.

3. RADIATION DAMAGE STUDIES

The studies of radiation damage in materials was one of the first applications of particle beams. Besides reactors, particle accelerators provided a means to determine the effect of radiation on materials used in the construction of equipment for a radiation environment. The studies today include tests of structural materials, of semiconductor devices, used in satellites or high flying airplanes and of metals, metal alloys and of insulators.

3.1 Radiation Effects in Metallic Materials

3.1.1 Search for first wall materials for fusion reactors

The investigation of materials which might be used in the first wall of a fusion reactor is of great interest and studied at many places. The simulation of the effect of 14 MeV neutrons, produced in the tritium-deuterium fusion reaction, on materials is needed to determine, if a specific material can be considered as first wall material. At PSI[17] a proton irradiation experimental area (PIREX) is set up especially to look at effects of protons on prospective materials. The proton beam in this area is degraded to 300 MeV and it can be degraded further in steps of 2.3 MeV to an energy as low as desired. The Institute for Advanced Materials of the European Communities in ISPRA is investigating materials for the first wall under irradiation and mechanical stress.[18][19] Similar experiments are also performed at VEC in Calcutta[20] and at the HIRFL in Lanzhou.[21] At KfK Karlsruhe a recently installed universal testing machine at their "dual beam facility" allows to investigate various static and dynamic loadings in terms of stresses and strains like push-pull fatigue, creep or creep-fatigue during irradiation in a vacuum chamber[22] (Fig. 2).

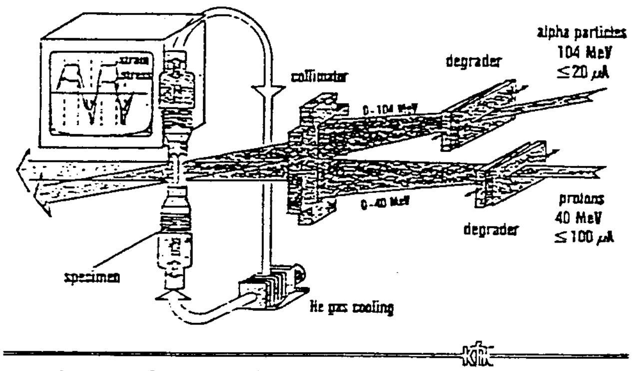

Figure 2 Scheme of the Karlsruhe dual beam facility.

3.1.2 Radiation effects on permanent magnets and amorphous metallic alloys

The effect of radiation on permanent magnets is looked into at NSCL in East Lansing[23] and at the cyclotron in Jyväskylä.[24][25] In both places a reduction of the magnetic field was observed.

The investigation of amorphous metallic glasses was pioneered by Klaumünzer et al.[26][27] and extended to higher energy beams by the group at GANIL lead by Jousset.[28] The original hope was to find a particular radiation resistant material. To the great surprise however a makroscopic growth of amorphous metals was observed under particle irradiation. The CIRIL group at GANIL has performed extensive studies of radiation effects on materials[29] over a wide range of energies available at the GANIL facility.

3.2 Radiation Effects on Semiconductor Devices

The effect of radiation on the performance and lifetime of semiconductor devices is of obvious importance for satellites and space probes. Accordingly experiments to simulate the radiation in space are done at many places, and test facilities to routinely check all devices are needed. One Single-Event-Upset (SEU) test facility is in operation at the tandem accelerator in Brookhaven,[30] and there are discussions about setting up a similar facility at VICKSI. At the Philips injector at PSI in Zürich an interesting simulation was caried out[31]: on the ESA Earth Resource Satellite

ERS-1 an instrument had failed after five days of operation. Immediately before the failure a transient overcurrent had been measured. Using the data of the on-board telemetry record, one could deduct, that a proton induced latch-up might have occured in a CMOS device. The tests at the OPTIS facility at PSI with a 60 MeV proton beam verified this conclusion beautifully.

3.3 Radiation Effects on optical Glasses

At INS Tokyo [32] the effect of proton irradiation on glasses was investigated, especially with the objective to determine the possibility to use these glasses for optical systems in a satellite. The transmission curves for optical glasses before and after 40 MeV proton irradiation are shown in Fig. 3. At

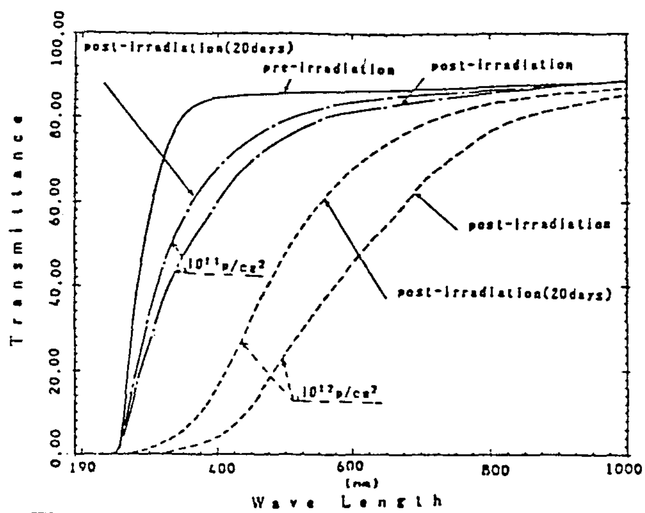

Figure 3 Transmission curves of optical glass before and after 40 MeV proton irradiation.

shorter wavelength the transmission is reduced considerably at proton fluxes above $10^{10}cm^{-2}$, but a significant recovery can be observed 20 days after the irradiation, indicating a self healing effect of the glasses. This might be similar to the production and bleaching out of "colour-centers" in alkali halides[33] which occurs especially at elevated temperatures. This effect is also important for glassfiber cables in a radiation environment as for instance accelerator tunnels.

4. BASIC RESEARCH IN SOLID STATE PHYSICS WITH PARTICLE BEAMS

The use of particle beams in solid state physics and in applied physics becomes more and more common. At VICKSI presently more than half of the beam time is used for experiments in these fields, with increasing demand. The purpose of the condensed matter studies is to get a better understanding of local properties like the magnetic behaviour of isolated atoms in metals depending on lattice sites or the dynamics of implanted atoms in solids. With the ion beam one produces shortlived radioactive isotopes and uses their recoil momentum to implant them into solids. The experimental setup for recoil implantation is shown in Fig. 4. In the upper part of the figur the primary ion beam will pass through the sample, while in the arrangement shown in the lower part the passage of primary ions through the sample is avoided. In the solid the nuclear moment of the recoil nucleus will interact with either the internal or an externally applied magnetic field, or the electric field gradient of the solid at the respective lattice site. These interactions can be observed by nuclear methods like the "Perturbed Angular Distribution Method" (PAD) or "In Beam Mössbauer spectroscopy". In the following these methods shall be given as examples.

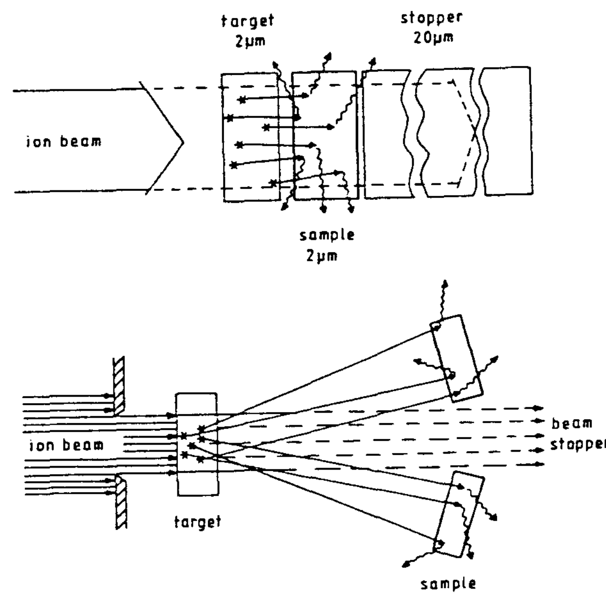

Figure 4 Experimental arrangements for recoil implantation.

4.1 Perturbed Angular Distribution

The measurement of the perturbed angular distribution is based on the fact, that the γ-ray emission of the recoil implanted radioactive isotope is modified by the interaction between the nuclear moment of the probe nucleus and magnetic fields or electric fieldgradients present at its location in the sample. The spin of the probe nucleus makes a Larmor-precession, and this precession is observed as a modulation of the γ-ray intensity in time. The period of this modulation gives information about the field or field gradients, provided one knows the magnetic moment of the probe nucleus.[34]

4.2 In Beam Mössbauer Spectroscopy

While the standard Mössbauer spectroscopy uses a long lived parent radioisotope, the in-beam method[35] uses a particle beam to excite the Mössbauer isotope via Coulomb excitation to the Mössbauer level. A ^{57}Fe target is bombarded with an ^{40}Ar beam at an energy well below the Coulomb barrier. This way any background from nuclear reactions is avoided. The Fe atoms are excited to the 14.3 keV Mössbauer level and simultaneously recoiled out of the target. The angular distribution of the recoils peaks between 20° and 70° with respect to the beam axis. So the probes can easily be implanted into the sample material avoiding that the beam strikes the sample. A sketch of an experimental

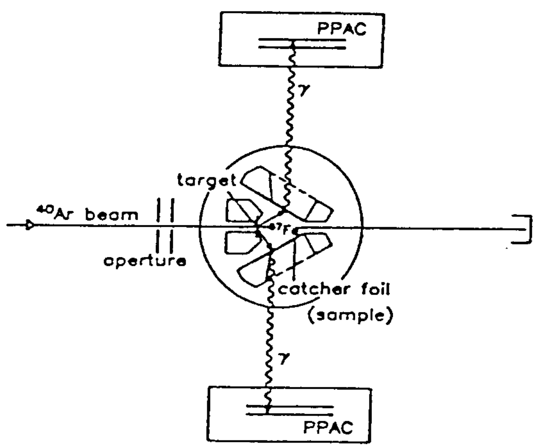

Figure 5 Sketch of the experimental setup for "in beam Mössbauer spectroscopy". PPAC stands for parallel plate avalanche counter.

setup for in-beam Mössbauer spectroscopy is shown in Fig. 5.

The actual measurements are taken between the cyclotron beam pulses, contrary to on-line Mössbauer spectroscopy, where a macropulsing of the cyclotron is required.

5. MATERIAL MODIFICATION WITH ION BEAMS

5.1 High Energy Implantation

While in the past implantation was performed at energies below about 500 keV, in recent years implantations at energies as high as several 100 MeV have been reported.[36] Low energy implantations have been used extensively to improve the surface behaviour of materials, reducing wear, friction, oxidation and corrosion,[37)38)] or to produce radiation enhanced cohesion[39)]. Implantations at high energies can be used to produce burried layers of ions in a solid, to produce insulating layers for instance in silicon by implanting with oxygen or nitrogen or to introduce doping atoms into semiconductor wafers. The applications are manyfold, but, to my knowledge, so far only used in experimental studies and not yet in industrial applications. This is partly due to the fact that high energy accelerators are not so easily accessible as low energy implanters but also because the users and operators of high energy facilities have not made much of an effort to introduce industry to the possibilities of their facilities.

5.2 Production of New Material Properties with Ion Beams

5.2.1 Microfilters

The production of microfilters with ion beams developed by Spohr and Fischer[40)]is since being pursued at many different laboratories. Besides GSI in Germany, Riken in Japan,[41)]also JAERI in Takasaki is producing these filters.[42)] FZR Rossendorf[43)44)] has also produced and marketed microfilters used in the food and beverage industry. They used a 30 MeV chlorine beam from their tandem accelerator. A particularly interesting development is

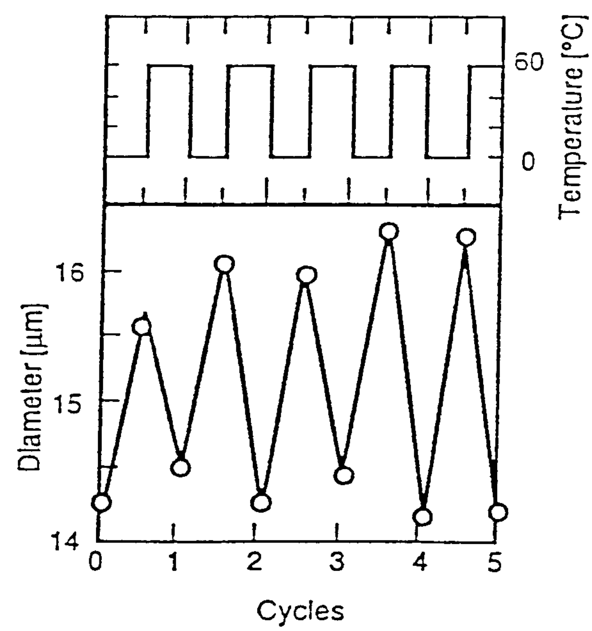

Figure 6 Change in size of pores formed in a thermally responsive film when cycled between 0°C and 60°C.

reported by Tamada et al..[45)] They produced a foil, which depending on the temperature was swelling or deswelling. When micropores were introduced into this foil, using heavy ion beams, the pore diameter could be varied between about 14 μ and 16 μ, depending on the temperature of the water the foil was immersed in. Fig. 6 shows how the pore size varies with temperature cycling. This is the first porous film, where pore size can be controlled by such an environmental condition as temperature. The filter is expected to separate various substances by changing the ambient temperature.

5.3 Influence of Radiation on High T_c Superconductor Properties

Very soon after the discovery of the high T_c superconducting ceramics the effect of radiation on the properties of these ceramics were studied.[46] The properties of the high T_c materials are affected by intrinsic defects and the structure-related anisotropy. The introduction of artificial defects by radiation can then be used to obtain a better understanding of the structural parameters and the related super- and normal-conducting properties of these materials. In general irradiation of high T_c materials lowers the critical temperature T_c. Therefore on first sight it appears, that radiation is harmfull for these materials. However extensive studies[47] using different projectiles have revealed, that the projectiles and the direction of the particle track with respect to the crystal axes can also have a positive effect on the superconducting properties.

5.3.1 Enhancement of critical current density J_c

High energy heavy ion irradiation using a 580 MeV $^{116}Sn^{16+}$ beam at the Holifield Facility in Oak Ridge has been shown to increase the critical current density J_c significantly.[48] Irradiations with protons or very light ions, which produce only point defects are not very effective in this respect. Irradiations with 770 MeV ^{129}Xe and 560 MeV ^{16}O done at the Texas A & M Cyclotron[49] and with 500 MeV ^{129}I at VICKSI[50] produced columnar defects which acted as flux pinning centers, and increased the critical current to values of 10^5 A/cm^2.

5.3.2 Radiation induced improvement of persistent field magnets using high T_c materials

At the Institute for Beam Particle Dynamics and the Texas Center for Superconductivity in Houston R. Weinstein et al. have produced permanent magnets by trapping magnetic fields in high T_c superconductors.[51] By irradiating the superconductors with protons at the Harvard Cyclotron, and with ^{3}He-ions at the Indiana University Cyclotron facility they could increase the trapped fields by almost a factor of four[52](see Fig. 7). Again this effect is caused by flux pinning. Using heavier ions and higher energies the trapped fields should increase even more. Persistent magnets with fields of 1.4 T have been produced and it is expected to reach 3 T this year and 6 T in the course of the next year. The possible applications of these magnets are manifold and very exciting: the production of external beam line magnets is in progress, a small motor using these magnets has been produced and the construction of a generator is being tested. Another application is the production of levitated bearings.

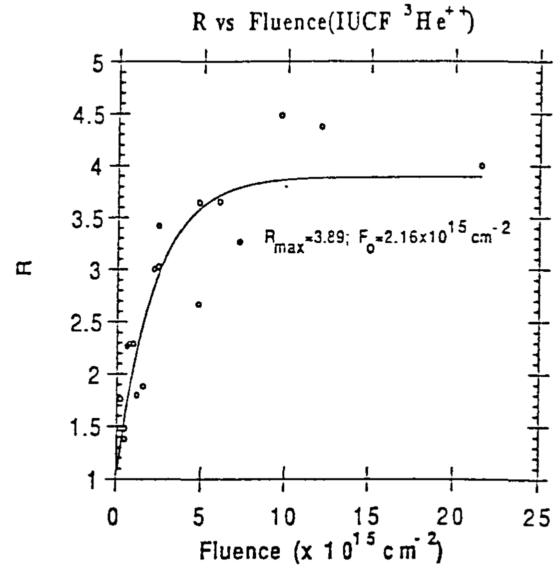

Figure 7 Ratio R of trapped field after to trapped field before irradiation versus particle fluence.

Many other applications are also considered. A very interesting application is the production of so called "replica" magnets.[53] Due to the pinning centers present in the superconductor the magnetic field is not expelled (Meissner-effect), but rather trapped. So superconductors with trapped field behave like metallic permanent magnets, except that they copy, essentially precisely, the fields which are used to activate them. So with one very precise prototype magnet one could reproduce identical magnets at will. The mechanical shape of the superconductor would not have to be precise at all, but nevertheless the field of the prototype magnet will be reproduced accurately. So far two technical problems have to be overcome: 1) It is still not easy to produce large volume high T_c material and 2) the trapped field magnets show a certain "creep", but there already exist ways to reduce this to only a few % per year and further improvements are certainly possible.

6. REQUIREMENTS FOR CYCLOTRON FACILITIES USED FOR MATERIALS RESEARCH

In the past most cyclotron facilities have been designed and built for the use in nuclear physics research. For material research however new requirements for beams and experimental equipment do become important.

6.1 Beam Properties

While for nuclear physics experiments small beam spots and a small emittance were the main requirements, in material research these beam properties might not be requested very often. At VICKSI we had situations where a 4" wafer needed to be homogeneously implanted with boron ions, and simple changes in the focussing did not produce a beam suitable for the given requirement. Ideally the experimenters would have liked four steering magnets to sweep the beam horizontally and vertically, and to ensure that the beam impinges on the surface perpendicularly. For implantations very high and constant intensities are required to be able to implant 10^{16} or 10^{17}ions/cm^3 in a reasonable time and with a high accuracy. For channeling experiments we were presented with the request to produce a parallel beam with a divergence of less than 0.01 mrad. Fortunately we have the space and a flexible enough beam optic system to produce this beam.
As the experiments in material research generally do not require very long times, quick energy- and ion-changes become important, and a special effort needs to be made to try to change the energy or the ion in a time small compared to the average irradiation time of a material research experiment.

6.2 Experimental Facilities

For surface investigations, implantations and analytical investigations often a very clean and good vacuum is required. Therefore ultrahigh vacuum chambers are needed. Often also vacuum manipulators are requested for the experiments. The control of the sample temperature from 4°K to as high as 1300°K is needed to perform the experiments in solid state physics. For the production of short lived PAC (perturbed angular correlation)-sources, which are recoil-implanted into the sample, remote handling and temperature control is needed to anneal the sample and eliminate the irradiation-produced defects. It is an interesting learning process which nuclear physicists and accelerator physicists have to go through, to understand all the requirements and experimental facilities which are necessary to perform high quality research in material science and in applications. It is obvious therefore, that considerable effort and money is necessary to supply all the required facilities. At VICKSI we are presently working on a proposal to provide an ion beam user-facility for solid state physics, material science and applications. One idea was to use an ECR-ion source injecting into a small cyclotron[54)], which then injects into the existing VICKSI-cyclotron. With no stripping between the source and the injector-cyclotron and the two cyclotrons, the intensity of VICKSI will increase significantly. Recently the development of frequency variable RFQ's[55)] has stimulated the idea to use one or two RFQ-accelerators instead of a small cyclotron. This would again give a large increase in intensity but a limited energy variation due to the restricted frequency variability of the RFQ's. Presently a layout which would give energies out of the cyclotron between 2 MeV/u and 6 MeV/u is being considered. This energy range is quite suitable for most "high energy" applications. The use of RFQ accelerators as injectors for VICKSI would hopefully furthermore decrease the required set-up time for a new beam.

7. ACKNOWLEDGEMENTS

I gratefully acknowledge the support I received from the cyclotron community. Over 90% of the laboratories I have addressed have responded and provided information about the research performed at their facilities. I am sorry that I was not able to use all the data I have been provided with, but time and space available made it necessary to make a selection which necessarily is biased by my personal preference. I apologize to all colleagues who might feel I have wrongly neglected their information. I am especially gratefull to all colleagues, who have granted permission to use their data in some cases even before publication. Finally I would like to thank the organizers of the Cyclotron Conference for giving me the opportunity to collect these data and present them at this Conference. I am sure I benefitted the most from it, and I am especially grateful for this.

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