

EPS Industrial Workshop: Towards Applications of Nano- and Quasi-Crystalline Materials

2-4 September, 1996, Berlin Germany

Scientific Report by H. J. Fecht, Co-Chairman of the Meeting

Summary

This was the first industrial workshop of its kind in the new and fast developing area of nanocrystalline materials. Whereas attention concentrated on nanocrystalline materials and their applications, new amorphous and quasicrystalline precursors were treated as well. The field of nanocrystalline or nanostructured materials is rather new and has created tremendous interest in the scientific community within the last decade. The impact of this new field can be judged by the large number of conferences and special symposia devoted to this new emerging field and the corresponding number of publications and new specialised journals.

In the meantime, there have been diverse successful examples for the application of these new materials in different industrial sectors worldwide. However, the full potential of these advanced materials has yet to develop and there are many opportunities in

- aerospace industry
- cutting tool industry
- energy technologies
- electronic engineering
- environmental engineering
- medical technology

Thus, the goal of this workshop was to give an overview of the

- synthesis
- characterization and
- properties of

nanocrystalline metals, alloys, ceramics and composites which can be produced either in the form of powders, thin films and coatings, or as bulk samples.

Participants and Venue

This international workshop was held in the "Magnus-Haus" of the German Physical Society in Berlin from 2-4 Sept-

ember, 1996. About 60 scientists and researchers from international research institutes, universities and industrial research laboratories participated in the scientific programme and lively discussion. The programme comprised 24 invited talks and a poster session with 11 posters of international contributions from: Canada; China; Germany; India; Israel; Italy; Japan; Russia; Slovakia; Switzerland; U.S.A. and U.K.

The strong industrial interest was documented by the large number of representatives from several different internationally operating companies, which for the most part were also integrated in the scientific programme, including:

- AEA Technologies
- Amorphous Technologies International
- Atotech
- Bayer AG
- Biotronik
- R. Bosch GmbH
- BMW Rolls-Royce
- Daimler Benz AG
- Engelhard Corp.
- HBI
- Merck KG
- Nanophase Technologies Corporation
- Nanodyne Corp.
- Nanometal Corp.
- Ontario Hydro Technologies
- Siemens AG
- H.C. Starck GmbH & Co.KG

Sponsors were: The European Commission, Directorate General XII; Science, Research and Development (Brussels, Belgium); and BMW Rolls Royce AeroEngines (Oberursel, Germany)

Scientific Programme

The scientific programme was organised into 8 different sessions plus a parallel poster session. The various sess-

ions consisted of 25 minute talks with ample time for discussion. The sessions were focused on the state of the art of the field and included:

1. Synthesis, processing and characterization
2. Bulk metallic glasses
3. Application of coatings
4. Structure - property relations
5. Diamond, DLC and SiC
6. Mechanical properties
7. Technology and applications
8. Technology transfer and general discussion

After a brief introduction by the organisers, **H.J. Fecht** and **H. Hahn, R. W. Siegel** from Rensselaer Polytechnic Institute, U.S.A., started out with:

Nanostructured Materials: A New Frontier. An overview of the field was given, discussing a wide variety of artificial nanostructured materials which have been synthesized and studied for more than a decade using a number of experimental methods. The structures and properties of these materials were elucidated in several important areas and fundamental understanding of the relationships among these areas is now beginning to unfold. Investigations of their chemical, electrical, magnetic, mechanical and optical behaviour have demonstrated the possibilities of engineering the properties of nanostructured materials through control of the sizes of their constituent clusters, grains or layers, and the manner in which these constituents are assembled.

R. Birringer from the University of Saarbrücken, Germany, discussed:

Relevant length scales in nanocrystalline materials and their property control. This is particularly important for the basic understanding of the magnetic and mechanical properties of nanocrystalline materials.

Focusing on the commercialisation of nanomaterials, **Romano** from Nanophase Technologies Inc., U.S.A., discussed:

Application opportunities for nanoparticles made from the condensation of physical vapours. As a consequence of world-wide research efforts devoted to the synthesis and processing of nanophase materials, a variety of commercial and industrial opportunities are now being developed for this new emerging technology. Although there are several synthesis methodologies, differentiation arises from the provision of controlled particle size and a weak degree of agglomeration, as well as manageable

surface chemistry. All of these attributes have been achieved through the condensation of physical vapours. Examples for the development of commercial products based on nanophase materials were presented: nearly net-shape formation of ceramic components and UV-absorbant TiO₂ nanoparticles for human skin protection, and other environmental issues. Nanophase Technologies is a small upcoming company, with currently 80 employees.

H. Wollenberger from the Hahn-Meitner-Institute Berlin, Germany, demonstrated new methods for:

Characterization of nanocrystals in amorphous matrices by means of AP-FIM and SANS. With these methods the size distribution, the growth behaviour, the chemical composition and the magnetization can be accurately determined. In particular, the nanoanalytical tomographic atom-probe in combination with field ion microscopy offers tremendous potential. For example, the local chemical composition of nanostructured superalloys for aerospace applications and the early crystallisation steps in bulk metallic glasses can be analyzed atom by atom, leading to precise control of the nanostructure during the preparation and usage of these advanced materials.

A. Inoue from Tohoku University, Sendai, Japan, continued with his contribution:

High-strength nanogranular quasi-crystalline alloys. In this new research effort on the production and use of bulk metallic glasses, he showed how high strength Al-based alloys have been developed through the formation of a novel structure of nanogranular icosahedra with fcc-type precipitates. The average grain size corresponds to 10 to 50 nm for the β -phase and about 10 nm for the Al phase. These alloys represent a broad class of metallic materials. They have excellent mechanical properties with a tensile strength of 1200 to 1400 MPa, corresponding to an increase by a factor of five to seven in comparison with conventional Al-alloys. Elongation can reach values of up to 30% with an impact fracture energy of 16 Jcm⁻². In Japan a broad research effort is dedicated to this new type of advanced material for lightweight structural components.

K. Lu from the Chinese Academy of Sciences, Shenyang, China, demonstrated an alternative synthesis route:

Nanocrystalline materials crystallised from amorphous solids: nanocrystallisation, structure and properties.

After crystallisation of amorphous precursors, the nanocrystalline materials exhibit some unique structural characteristics and novel properties which are fundamentally different from those of conventional coarse-grained polycrystalline materials with the same average chemical composition.

Subsequently, applications of nanocrystalline materials as a coating material were discussed. **N. Kanani** from Atotech Berlin, Germany, illustrated the use of:

SPM as a means of studying the nucleation process and crystal growth modes. *In-situ* scanning probe microscopy (STM/AFM) is becoming an increasingly valuable technique for examining the quality and performance of printed circuit boards. In particular, this method has been used for *in-situ* studies of the palladium activation process for applying electrodeless copper on epoxy resins. The classification of growth modes in the early nanocrystalline regime in the absence and in the presence of organic additives was discussed in terms of thermodynamic models which predict the growth morphology as a consequence of deposit-substrate interaction and strain arising from lattice misfits.

T. Haubold from BMW Rolls-Royce, Oberursel, Germany, gave an overview on:

Requirements for nanostructured coatings in aeroengines. The main applications here are wear-resistant coatings, oxidation and corrosion-resistant coatings, thermal barrier coatings, as well as abrasion-resistant coatings. Techniques to apply the coatings are diffusion processes, thermal spraying or EB-PVD. However, certain barriers have to be overcome in order to be able to use nanocrystalline coatings and gradient nanostructures with a sufficient service life-time. Here, adequate stabilization of the nanostructure and defect structure against coarsening and sintering at the high service temperatures is essential in order to prevent change in the tailored properties in conjunction with special substrate requirements.

In the field of biomedical materials **Eck** from Biotronik, Germany, discussed the use of:

Coatings for medical applications. For body stents, electrodes for pacemakers, fault current limiters etc., coatings compatible with the human body's chemistry for an extended period of time become necessary. Here, research is focused on

amorphous and/or nanocrystalline layers of SiC, TiN and more complex oxides and nitrides.

To follow, **B. Straehler** from Siemens, Berlin, Germany, presented a survey of:

The increasing importance of coatings for the electrical industry. Even outside the fields of semiconductors and displays, the number of applications for coatings and, in particular, nanocrystalline coatings in the electrical industry is steadily growing. This is mainly due to the miniaturization and multifunctionality of products which enforce a distinction between bulk and surface properties. This has been shown for a number of applications encompassing surface functions, such as electromagnetic shielding, electrical (super) conductivity, biocompatibility, wear and corrosion resistance. In addition, dry machining and the forming of metals with coated tools are of great importance not only for environmental but also for productivity reasons, because integration of these processes in production lines will be possible. Examples were discussed, such as metal-carbon coatings by magnetron sputtering, diamond coatings by microwave assisted CVD and cubic boron nitride coating by cathodic arc evaporation.

The following day began with a contribution by **B. Cantor** from Oxford University, U.K., titled:

The effect of processing conditions on the structure of nanocomposite materials for a variety of different applications. His talk described detailed transmission electron microscope studies of the microstructure of a variety of nanocomposite materials in conjunction with the optimization of structure/property relationships. The materials discussed included devitrified amorphous Fe and Al nanocomposites for high-strength/high-modulus aerospace applications; rapidly solidified immiscible Al-Pb and Al-Sn nanocomposites for journal bearing and other low friction applications, and co-sputtered Ag-SiO₂, Ag-Si and Ag-ZnO nanocomposites for optoelectronic and diffraction grating applications.

Then **U. Erb**, now at the University of Toronto, Canada, gave an overview on:

The structure, properties and applications of electrodeposited nanocrystals. He demonstrated how electrodeposition can be a technologically and economically viable synthesis route to produce nanocrystalline metals, alloys and composites, both in bulk form and as coatings. Improvements in hardness and corrosion resistance (as grain size dependent

properties) and thermal expansion and saturation magnetization (as grain size independent properties) have been achieved. In the subsequent discussion industrial applications leading to new business opportunities for several industries were elucidated for corrosion and wear resistant coatings, heat exchanger repair, magnetic data storage and electrical connectors (see G. Polumbo).

A.L. Greer from Cambridge University, U.K., presented his contribution on:

Hardness and abrasive wear resistance of Al-based nanophase composites made by devitrification. These new aluminum/transition-metal/lanthanide alloys are of interest primarily for their mechanical properties, notably their exceptional (for Al alloys) specific tensile strength. The formation of α -Al nano-crystallites during devitrification annealing is accompanied by a significant increase in microhardness, so that values equivalent to a yield stress as high as 1.6 GPa can be attained. Coarsening of the nanophase microstructures is rather slow and is accompanied by an increase in the volume fraction of α -Al, apparently associated with Gibbs-Thomson effects altering the metastable equilibrium matrix composition for small particles. The abrasive wear resistance of the materials has been measured using a micro-scale test. As expected for materials of such high hardness, the wear resistance is good, and indeed exceptional for Al alloys.

To follow, **K. Niihara** from the Institute of Scientific and Industrial Research, Osaka University, Japan, gave an overview on:

The fabrication processes and mechanical properties of ceramic-based nanocomposites. Ceramic-based nanocomposites in which nano-sized ceramics and metals are dispersed within matrix grains and/or at grain boundaries, were successfully fabricated in the $\text{Al}_2\text{O}_3/\text{SiC}$, $\text{Al}_2\text{O}_3/\text{Si}_3\text{N}_4$, $\text{Al}_2\text{O}_3/\text{TiC}$, $\text{Al}_2\text{O}_3/\text{TiB}_2$, MgO/SiC , $\text{ZrO}_2/\text{SiCMoSi}_3$, $\text{B}_4\text{C}/\text{TiB}_2$, Sialon/SiC , $\text{Si}_3\text{N}_4/\text{SiC}$, $\text{Si}_3\text{N}_4/\text{BN}$, Sialon/BN , $\text{Al}_2\text{O}_3/\text{W}$, $\text{Al}_2\text{O}_3/\text{Mo}$, $\text{Al}_2\text{O}_3/\text{Fe}$, $\text{Al}_2\text{O}_3/\text{Ni}$, ZrO_2/Ni , ZrO_2/Mo composite systems by conventional powder metallurgy methods. The striking feature of these nanocomposites is that the mechanical properties are significantly improved by the nanometer-sized dispersion from 5 to 10 vol%, even at high temperatures. For example, fracture strength and creep resistance in the ceramic/ceramic composite systems were improved by 2 to 5 times and by 3 to 4

orders of magnitude, respectively, depending on the composite system. The important role of intragranular and intergranular nano-dispersions in the improvement of mechanical properties was further discussed.

B.H. Kear from Rutgers University, New Brunswick, U.S.A., then presented new developments in:

Spray deposited nanostructures: a new generation of high-performance coatings. He has investigated two opportunity areas for thermal spray deposition of nano-structured coatings: (1) ambient pressure deposition, using conventional plasma arc (PA) and high velocity oxy-fuel (HVOF) processes, and (2) low pressure deposition, using a novel low pressure combustion flame (LPCF) process. Both types of coating treatment have numerous applications. These are well known for conventional thermal spraying, since the technology has undergone more or less continuous development over the last two decades. In contrast, applications for LPCF technologies are only now being recognized. The main advantage of the new technology is its capability for uniform deposition over large areas. Recent developments in this field were further discussed, aiming to realize high-quality spray deposited nanophase coatings.

A further talk by **J.H. Perepezko** from the University of Wisconsin-Madison, U.S.A., focused on:

Processing of nanocrystalline materials: Some issues in surface coatings and films. Numerous strategies involving vapour phase, solidification and solid state processing are available in order to produce nano-structured materials in the form of thin film coatings. Often technological applications proceed on an empirical route, but an examination of the methods reveals a number of common kinetic and thermodynamic features. For surface coatings and films which offer attractive industrial applications, nanocrystalline products require limiting the growth of existing phases. With suppressed growth, surface processing with energized beams or depositions elevates the free energy to expose metastable and non-equilibrium states. The general basic issues concerning the kinetic competition controlling nanocrystal development and stability has been illustrated on the basis of experience from laser surface modification and devitrification reactions of amorphous precursors.

In the following session on diamond

and diamond related nanomaterials **C.P. Klages** from the Fraunhofer Institute for coating technologies, Braunschweig, Germany, discussed:

Diamond-like carbon coatings, basic properties and applications. The presentation gave an overview on pure amorphous carbon (α -C), hydrogenated amorphous carbon (α -C:H), α -C:H containing nanosized metals or metal carbides and network modified α -C:H containing hetero-elements like F, Si and N. The mechanical-tribological properties of these coatings which form the basis of most of the present day's applications, are characterized by small friction coefficients against metals and hardness values which are typical for ceramic materials, combined with polymer-like properties, such as high elasticity and low surface energies. Several examples have been given in which diamond-like carbon coatings have successfully been applied in tribological systems.

In a joint venture between **M. Werner** from VDI/VDE-IT, Teltow, Germany, and **C. Johnston** from AEA Technologies, Oxford, U.K., **M. Werner** presented the recent findings on:

Properties and advanced applications of CVD-diamond. Diamond is the ultimately hardest material known. It has the highest thermal conductivity at room temperature and excellent mechanical properties. Undoped diamond is an insulator whereas B-doped diamond becomes metallic. These properties can be tailored further by the control of grain boundaries in nano- and microcrystalline CVD diamond. The control of the thermal and electronic properties has been described in detail. Near future applications of small grained CVD diamond as basic material for heatspreaders and sensors have been discussed.

Then **G. Krötz** from Daimler Benz AG, München, Germany, gave an overview on:

SiC thin films in the amorphous, nano-, poly- and single-crystalline state: Deposition methods, properties and applications. SiC in its different states has a number of extraordinary properties compared with other semiconductor materials. It has a large, and in the case of the amorphous state, variable band gap, high resistance to chemically aggressive media, high thermal conductivity, good high temperature elasticity and high hardness. These properties make SiC attractive for optical applications especially in the UV range and for sensors operating in harsh environments. The

effect of internal interfaces on the properties is evident and can be controlled by different (ion beam assisted) deposition methods. The potential of these films and several examples of applications have been discussed.

On the last day of the meeting **R. Rosen** from the Technion in Haifa, Israel, started with his overview on:

Cemented Carbides from micron to nano grain size. Sintering behaviour, microstructure and mechanical properties of WC based composites have been studied for nanocrystalline and submicron sized powder products and compared with conventional micron sized powder products. A grain size reduction generally improves the hardness, fracture toughness and transverse rupture strength as well as the solid-state consolidation with a lower activation energy and higher final density. These improvements lead to new designs and applications in the hard metal tool industry.

Then **E. Bonetti** from University of Bologna, Italy, discussed:

Mechanical spectroscopy in the study of nanophase materials. These measurements in the range 3-3000 Hz on nanostructured Fe, Al, Fe-Al and FeCuSiB alloys prepared by mechanical alloying and rapid quenching were reported using an inverted torsion pendulum and a vibrating reed apparatus. Intrinsic mechanisms, such as grain boundary or interface relaxation, damping effects related to a reduced linear defect density, high temperature superplasticity and magnetoelastic phenomena are observed.

In the session of Technology and Applications **T. Mehlhorn** from Siemens AG in Berlin, Germany, presented:

New developments in electronic

applications. Here, he showed new ways of designing circuit boards for high temperature applications and advanced cooling systems for laser diodes. The most promising design is based on anodized aluminium technologies with a special nanostructured surface layer.

In the following, **A. Dommann** from the Institute of Microsystem-Technology in Buchs, Switzerland, presented:

Amorphous metals as new materials for transducer applications. The amorphous microstructure of sputtered TaSiN thin films can hamper or impede fatigue effects typical for coarse-grained materials. This can lead to applications in micro-electro-mechanical devices, such as sensors and actuators. The fabrication using sacrificial layers technology leads to free standing amorphous films with the desired electrical and mechanical properties matched to Si substrates.

Then **R.D. Shull** of the National Institute of Standards and Technology, Gaithersburg, U.S.A., discussed:

Magnetic applications of nanocrystalline and nanocomposite materials. New magnetic phenomena, unusual property combinations, and both enhanced and diminished magnetic property values are some of the changes observed in nano-crystalline matter from conventional magnetic materials. Exciting applications are permanent magnets, soft ferro-magnets, magnetic field sensors, magnetic recording media and magnetic refrigerants. A review of the magnetic property changes was given emphasizing the reduction of some critical magnetic length scales to nanometer dimensions.

G. Polumbo from Ontario Hydro Technologies, Toronto, Canada, presented the role of:

Nanocrystalline nickel deposition for steam generator repair as an important example for the application of nanostructured materials in Environmental and Energy Technologies. Based on the fundamental work on electro-deposition of nanocrystalline nickel presented by Erb (see above) a technology for on-line repair of heat exchangers in nuclear power plants has been developed. The combination of corrosion resistance, mechanical properties and thermal stability of the nanostructures are essential for this application. In Canada and the U.S., this method has been successfully employed resulting in immense reductions of repair cost due to reduced shut-down times.

In a final session and intense discussion on Technology Transfer **Kramer** from the German Ministry of Science and Technology, Bonn, gave an introduction on:

Industrial applications of results from nanotechnology-related R&D- Technology transfer in Germany. During his presentation and following discussions a strong need for a new R&D program on nanotechnologies and nanostructured materials has been expressed. The further discussion included the various routes of technology transfer leading to "incubators" and the initiation of small businesses. In particular, the difference in the financial schemes and medium term business perspectives in the U.S.A. and Europe seems to explain the divergence in the number of small high-tech businesses developed by technology transfer from basic research. In this regard, nanostructured materials are considered worldwide as a growing opportunity for the development of advanced products in a variety of industries.

Women in Physics Lecture Tour

Australian Institute of Physics

In 1998, the Australian Institute of Physics will sponsor a Woman in Physics Lecture Tour in recognition of the contribution of women to advances in physics. The lecturer will be a woman who has made a significant contribution in a field of physics research and has demonstrated public speaking ability. She will give a lecture in Canberra and in each of the six Australian State capital cities during a 2-3 week period between May and August. There will also be

opportunities to visit universities and other scientific centres throughout Australia to give research colloquia where appropriate. Air travel to and within Australia, and accommodation will be provided. We seek your assistance in bringing this opportunity to the attention of a wide range of women physicists. Readers who are interested in making a nomination for this lecture tour should contact:

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If you require more information then please contact Dr. Margaret Law or Dr. Judith Pollard. We would supply a notice, to appear in "The Physicist" the Australian Institute of Physics magazine, which contains more detailed information about the lecture tour and the nomination procedures.
Closing: 15.07.97

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