

Project no. CA 505549-1 Project acronym DESHNAF

# Super-Hard Nano-Composite films by plasma processing

Instrument: Coordination Action
Thematic Priority: NMP

# **Final Publishable Activity Report**

Period covered:	from 1.2.2004	to 31.12.2006	Date of preparation 15.2.2007		
Start date of project: 1.2.2004			Duration: 35 months		
Project coordinator name: Dr. Ilana Fried					
Project coordinator organisation name: Euro-Consultants (Israel) Ltd.			Revision: Draft 1		



Figure 1 International Conference Picture - Ein Geddi

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# The Project

Deshnaf is a Coordination Action supported by the NMP Priority of FP6. the consortium included eight members from Germany, UK and Israel and an Industrial Advisory board from Austria (Plansee), Switzerland (Platit) and Israel (Iscar).

Deshnaf is about super-hard materials. In particular it is interested in nanostructured super-hard materials, priority given to plasma deposited coatings.

Super-hard materials films (> 40 GPa) are useful in several sectors of the Industry: *machine tools, engine parts, gas turbine blades, etc.* They are needed wherever rapidly moving parts come into contact with hot, corrosive environment or when high friction is created. The most efficient way to make such super-hard materials is by plasma activated techniques using chemical vapour deposition (PACVD), physical vapour depositions (PAPVD) and mixtures of the two methods.

It turns out that composite materials, which include a nano-structured phase, exhibit the highly desirable properties: high hardness and wear resistance at elevated temperatures. However, these very new technologies exhibit some problems which DESHNAF is proposed to help solve.

Two families of super-hard materials are considered: a group containing *two hard phases* (for example:  $nc^1$ -TiN and a-Si<sub>3</sub>N<sub>4</sub>): nc-MeN/a-Si<sub>3</sub>N<sub>4</sub>, a-TiSi and a group containing *one hard phase and a second soft phase* (for example: ncZrN+Cu), nc-MeN/Metal (e.g. Cu, Fe, Ni, Y, Mo, Ag, Co).

# **Objectives**

The objective of DESHNAF is: to coordinate fundamental knowledge and the development of generic technologies with a broad range of applications in many industrial sectors, mainly machine tools, by addressing the following topics:

- 1. Correlation of plasma and deposition variables with the deposition process
- 2. Measurement of super-hardness, especially on very thin, nano-composite materials
- 3. The stability of the films and their resistance to oxidation and wear at high temperatures
- 4. The tribological properties of super-hard, nano-composite films
- 5. Transfer of technology between Industry and Academia
- 6. Exchange of personnel to foster cooperation and learning
- 7. Dissemination of results of research, and insights gleaned from the project
- 8. And last, but not least: foster collaboration among the partners of the consortium themselves and others working in similar areas

# The objectives fulfilled

In the following, it will be shown how the consortium fulfilled the objectives of DESHNAF. The numbers of the sections are the same as the number given to the objective above.

1. Correlation of plasma and deposition variables with the deposition process

<sup>&</sup>lt;sup>1</sup> nc- and a- denotes the nanocrystalline and amorphous phases respectively and MeN denotes the nitride of a metal (Ti, Zr, Hf, W, V, Cr, etc.).

A workshop was conducted by DLR Stuttgart and a literature review was created by IPP Garsching on the influence of experimental variables on the resulting coating.

#### Result:

- Several plasma deposition methods bring good results: PVD, PACVD. Good results are also obtained by using mixtures of those two methods. Plasma spray, which belongs to a different class of plasma technologies, is also suitable for the production of super-hard, nano-composite coatings.
- The PVD methods have developed into mature tools for the deposition of super-hard nano-composite coatings.
- The study created a knowledge base allowing the matching of properties of coatings to suitable deposition methods

# 2. Measurement of super-hardness, especially on very thin, nanocomposite materials

A workshop devoted entirely to the measurement of super-hardness was set up by TUM (Garsching). Since this is a topic of great concern to many scientists and engineers working in the field, discussions were carries out also at other DEAHNAF gatherings

Result: a close exchange of fundamental knowledge and experience between academia and experts from the industry, particularly from companies producing the equipment for measuring hardness on very thin films

# 3. The stability of the films and their resistance to oxidation and wear at high temperatures

Both a workshop and a literature review on the stability of super-hard films and their resistance to oxidation and wear at high temperatures were conducted. The workshop was coordinated by BGU and the literature review was carried out jointly by ECIL and BGU.

Result: Nano-composite super-hard materials are the materials of choice for high temperature stability and resistance to oxidation. The only other material fulfilling these two criteria simultaneously is cubic BN

## 4. The tribological properties of super-hard, nano-composite films

A workshop was conducted on the tribological properties, methods and instruments for measurement of material properties under various conditions of use. Another topic was the prediction, based on theoretical knowledge, of expected tribological properties of materials. The workshop was conducted by the University of Sheffield.

Result: The most important feature for high tribological properties is the quotient of the hardness coefficient and Young's modulus. Super-hardness is not so important for best resilience and low friction.

## 5. Transfer of technology between Industry and Academia

The international conference and the last workshop enabled the free discussion of problems, solutions and methodologies among all participants from both Industry and Academia.

Result: Cross fertilization of minds, exchange of ideas and new avenues for collaboration. These are manifested by the intention to submit joint proposals to FP7, as well as other forms of collaboration. It is interesting to note that the transfer of ideas and knowledge took place from industry to academia as well as from academia to industry.

#### 6. Exchange of personnel to foster cooperation and learning

Exchange of personnel took the form of visits, short of longer duration, advanced courses. Details are given in the sections devoted to each partner. The following table is a short summary of the exchanges that took place.

Institution	Received visitors from	Sent visitors to
TUM	BGU	USFD
TAU	USUR, USFD, Plansee, research institutes in Russia and the Ukraine	USUR
USFD	TUM, Univ. Cyprus, Moscow State Univ.	TAU
IPP		USUR
USUR	IPP, TAU, USFD, BGU, Univ. Cyprus	TAU, ENEA in Brindisi
BGU	Univ. Lodz; Univ. Montreal	TUM, USUR

#### Result:

- Courses in materials engineering of USUR were of great value for DESHNAF partners
- Overall experience was very good in all exchanges

# 7. Dissemination of results of research, and insights gleaned from the project

Dissemination took the following forms:

- The DESHNAF web site (www.deshnaf.net) in a source of much information about the project. It includes the reports of all the workshops, full text of the two studies and also publications of other research (see screenshots on the following page). In addition, the web site includes a forum, links to the partners' web sites and news.
- We conducted a mail-shot to East Europe and other countries informing a large number of researchers of the project. This was done twice during the project: towards the end of the first year and in the third year.
- The publicity created by the international conference was a very powerful dissemination action and brought many to the conference itself. The publication of most of the presentations in the prestigious journal Surface and Coating Technology adds to the publication of DESHNAF among researchers in the field.
- Participation of DESHNAF partners in international conferences was an important tool for dissemination. Details are presented in the sections devoted to each partner. In addition, some partners were invited to give seminars in European institutions.

# 8. foster collaboration among the partners of the consortium themselves and others working in similar areas

Collaboration in research:

- Two research proposals were submitted to the last NMP Call of FP6: Plasdofil and RIHPEN
- The consortium wishes to submit other proposals for research and/or support-coordination projects in order to exploit the results of DESHNAF and enhance the collaboration reached so far. Four or five proposals are planned for the first Call of FP7.

# Other collaboration:

• Collaboration in testing and analysis is planned

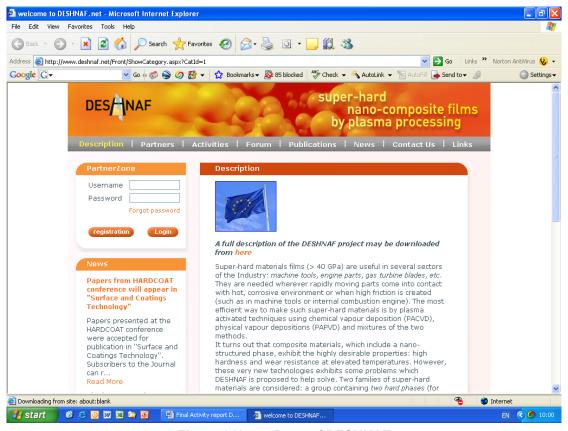


Figure 2 Home Page of DESHNAF

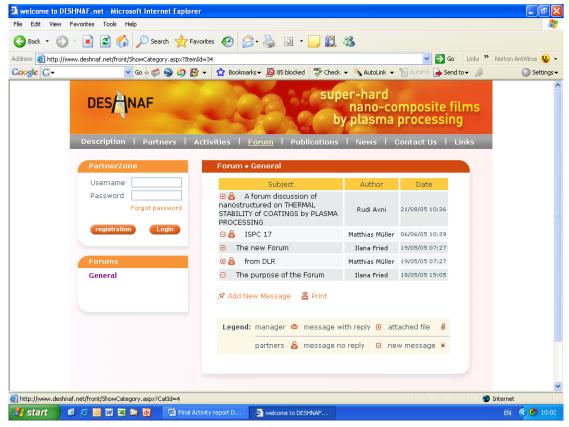


Figure 3 Forum page of DESHNAF

 Consultation of researchers from the Academia to help solve industrial problems is also planned.

Collaboration in research other than through FP7 is envisaged.

# **Impact**

The impact of DESHNAF was as great as we could expect in terms of interest the project and its activities have raised. The impact influences several circles: those attending the functions of DESHNAF, the visitors to the web site, the readers of the various publications and the attendants of the conferences where the partners of DESHNAF lectured. However, in assessing the impact, only those who participated actively in the various activities of DESHNAF are considered:

- DESHNAF reached researchers in 23 countries spanning all continents
- It had impact on the Industry: at least 5 large companies as well as forward looking 8 SMEs
- A large number of researchers from the Academia and research communities participated in its activities (over 100 researchers from over 20 institutions)
- This large and diverse participation opened new avenues for collaboration in research

In addition, DESHNAF had impact on science:

- Theories
  - Development of Differential Thermodynamics theories from first principles, as well as understanding of the thermodynamics of the phase segregation needed for the formation of super-hard nanocomposites
  - Understanding of the importance of H/E for good tribological properties
- Practice
  - Understanding of the experimental conditions needed for achieving nano-structured super-hardness
- Implementation
  - Close collaboration with industry and transfer of the basic knowhow by TUM and others

## **Deliverables**

# D2.1 Plasma Techniques - Workshop

**Date**: 20-22 September 2004 **Place**: Stuttgart, Germany

Attendance: 33 people from 17 companies or institutes in 7 European countries

Scientific level: High

**Results**: Discussion of various methods of plasma deposition

## D3.1 Study: Plasma variables and deposition processes

Compiled by D. Levchuk and H. Maier

IPP Garsching, Germany

Published on the DESHNAF web site

Scientific Level: Very high

## D4.1 Hardness measurement – Workshop

**Date**: 9 – 11 November, 2004 **Place**: Garching, Germany

Attendance: 28 people from 15 companies or institutes in 5 European countries

Scientific level: Very high

Results: Significant improvement of the understanding of the problems and how

to solve them

## D5.1 Thermal Stability and Oxidation Resistance – Workshop

Date: 21-23 September 2005

Place: Eilat, Israel

Attendance: 30 people from 12 companies or institutes in 5 European countries

Scientific level: High

**Results**: Understanding of the fundamental causes of thermal stability and

resistance to wear at high temperatures

# D5.1 Study: Thermal Stability and wear resistance

Compiled by: R. Avni, I. Fried, I. Zukerman, A. Raveh

Ramat Gan and Be'er Sheva, Israel

**Published** on the DESHNAF web site and of an article in Surface and Coating

Technology

Scientific Level: Very high

# D7.1 Tribology - Workshop

Date: 11-13 January 2006

Place: Sheffield, UK

Attendance: 27 people from 10 companies or institutes in 5 European countries

Scientific level: high

Results: the importance of the relationship between hardness and elasticity as a

predictor of good tribological properties

## **D8.1 International Conference**

Date: 27 February - 1 March 2006

Place: Ein Gedi, Israel

Attendance: over 120 people from 24 countries of all 5 continents; 38 lectures

and 19 posters

Scientific level: very high

#### Results:

- Exchange of information among a large number of researchers
- An opportunity for researchers from less advanced countries or institutes to present their work
- Papers will be published in Surface and Coating Technology after the customary peer review. (see announcement in the following page)
- The decision to extend the duration of DESHNAF and create the workshop on applications of super-hard, nano-composite coatings

# D9.1 web site www.deshnaf.net

#### Includes:

Project description

All public deliverables

Publications by members of the consortium

Forum

Links, including links to other professional web sites.

# D10.1 Industrial Applications – workshop and seminar

Date: 25 - 28 October 2006

Place: Prague, Czech Republic

Attendance: 38 people from 28 companies or institutes in 11 countries

Scientific level: Very high, participation of leading companies

**Results**: Very active discussion, high impact on the attendees from academia and SMEs, open and frank exchange among professors and researchers from

Industry.

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### Preface

This special issue of Surface and Coatings Technology on Superhard Coatings is based on papers selected from presentations at the International Conference on Superhard Coatings, held from February 27 to March 1, 2006 in Kibbutz Ein-Gedi, Israel. The conference was the culminating activity by "DESHNAF", a consortium and coordination action in the framework of the European Community 6th Framework on the topic of superhard nanocomposite films by plasma processing. The complete conference program can be found at the conference website, www.hardcoat.org. Sessions included:

- Interfaces, impurities and contaminants in the substrate/ coatings region
- Techniques for depositing superhard and nano-structured coatings
- Characterization techniques for superhard and nano-structured coatings
- Thermal stability and properties of superhard and nanostructured coatings
- Tribology and mechanical properties of superhard and nanostructured coatings
- Industrial applications of superhard and nano-structured coatings.

The General Conference Chair was Professor Raymond Boxman from the School of Electrical Engineering, Tel-Aviv University, Israel. The editors thank the conference sponsors for their support of the conference and this special issue: DESHNAF, GM Foundation, Iscar Ltd., Israel Scientific Instruments Ltd., Israel Plasma Science and Technology Association, and Tel-Aviv University — Faculty of Engineering. The editors also thank all of the conference session chairmen, the members of the conference organizing committee, and especially Program Chair Professor Rudi Avni, who is also the DESHNAF coordinator, Conference Secretary Alexey Shashurin and Assistant Editor Dmitry Grach. Finally, the editors would like to thank all of the referees for their efforts and timely submission of quality reviews.

Special Issue Editors
Vladimir Zhitomirsky
Tel-Aviv University, Faculty of Engineering,
Department of Interdisciplinary Studies,
POB 39040, Tel-Aviv 69978, Israel
E-mail address: zhitom@eng tsu.ac.i
Tel.: +972 3 640 8176,
fax: +972 3 641 0179.
Corresponding author.

Avi Raveh
NRC—Negev, Division of Chemistry, POB 9001,
Beer-Sheva 84190, Israel
E-mail address: aviraveh@hotmail.com.
Tel.: +972 8 656 8075;
fax: +972 8 656 8686.

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Figure 4 Anouncing the special issue of Surface and Coating Technology

# Partners' activities by partner

# Euro-Consultants (Israel) Ltd.

#### **Coordinator's Activities - Administrative**

- Preparation of the report of the all reporting periods, including gathering the information from all partners
- Managing the resignation of the University of Salford and introduction of Surrey, including amendment of contract
- Writing the request for the prolongation of DESHNAF, obtaining the consent of partners to have the additional workshop without adding to the expense of the project
- Maintenance of the web site, especially by adding the public deliverables and ensuring its continued presence on the air
- Collecting, writing and editing all deliverables
- Assembling the Industrial Advisory Board
- Creating the database for Mailshot to East Europe
- Organising four consortium meetings
  - > Kick off meeting in Brussels
  - > meeting in Eilat
  - > Meeting in Sheffield
  - > Final "Wrap up" meeting in Brussels
- Helping the partners in arranging the conferences and workshops
  - Coordination of WS1 in Stuttgart
  - Coordination of WS3 in Eilat
  - > Help to implement WS4 in Sheffield
  - Decision and support for WS5 in Prague
- Participation in the Organising Committee for the Conference in Ein Gedi

#### Coordinator's Activities - Professional

- Thermal Stability of TiAlBN and TiN/TiCN Coatings St.Petersburg HTPP 9
   Conference
- Plasma Surface Interactions in Plasma Deposition of Nanocomposite Hard Coatings Seminar at RWTH Aachen Germany
- Thermal Stability of Hard and Superhard Coatings, Seminar at Fraunhofer Inst Dresden Germany
- Invited to present lecture at Thin Films 2006, Singapore, 11-15 December.
   Title: Plasma Surface Interactions
- Participation in the Program Committee for the Conference in Ein Gedi
- responsibility for the Scientific Program
- Responsibility for the study on thermal stability of super-hard and related coatings. The study was done in collaboration with Ben Gurion University and included monolayers and super-lattice types of hard and super-hard coatings in addition to nano-composites.

# **Technical University Munich**

The **objectives** of TUM in DESHNAF was to contribute to all activities and functions of the project, especially in the areas of theoretical understanding, measurement of super-hardness technology transfer and dissemination.

#### Contributions from TUM:

- 1. Correlation of deposition variables with the superhardness:
  - Importance of the deposition temperature and nitrogen activity: Surf.
     Coat. Technol. 200 (2006) 3876 & Invited Review in Thin Solid Films 476 (2005) 1
  - Detrimental effect of impurities: J. Vac. Sci. Technol. B <u>23 (2005)</u> L 17;
  - ➤ Theoretical Understanding *ab initio* DFT calculations (Phys. Rev. Lett. <u>97</u> (2006) 086102):
- 2. Measurement of superhardness of thin coatings
  - Workshop at TUM see report of the Coordinator (well attended by participants from academia as well as from industry)
  - Our Work: Several papers, load-invariant hardness & verification by calibrated SEM:
- 3. Thermal Stability and Oxidation Resistance at elevated T
  - Significant Improvement of the Life Time of Coated Tools. Example: Dry drilling with tool made of cemented carbide coated with nc-(Al1-xTix)N/a-Si3N4 SHM (CZ) & PLATIT (CH)
- 4. Tribology:
  - Coefficient of friction of 0.6 0.7 at RT (0.4 for TiN) but it decreases with increasing T for the nanocomposites whereas it increases for TiN Published in: S. Ma et al., Surf. Coat. Technol. <u>194</u> (2005) 143.
- 5. Transfer of technology between Academia and Industry by collaboration with companies
  - SHM (Czech Republic)
  - Platit (Switzerland)
  - ➤ UNIMERCO (DK & GB)
  - Several SME's
- 6. Workshop on Industrial Applications, Prague October 2006 by DESHNAF and MACHERENA
- 7. Exchange of personnel and collaboration:
  - > Dr. I. Brunnel (TUM) visited Univ. Sheffield
  - > Dr. D. Kurapov (TUM) visited Univ. of Brussels
  - > Mr. I. Zukerman (Ben Gurion University) visited TUM
  - Many very useful discussion with the DESHNAF Partners during Workshops & meetings;
  - A proposal for EU 7th Program is in preparation in collaboration with TUM & Univ. Surrey, Sheffield, Linköping, Cyprus, and industrial companies SHM (CZ) and UNIMERCO (DK)
  - > DESHNAF Follow-up project: Dissemination of the industrial applications to SME's: There is indeed large need for that !!!

#### 7. Dissemination:

> TUM published during the project period more than 25 papers in the field of superhard nanocomposites

Many Invited and Plenary Lectures at International Conferences (only since January 1st, 2005 18 such lectures.

Emphasis on the Issue of Reproducibility of the Preparation of Nanocomposites with Hardness ≥ 50 GPa

# Deutsches Zentrum für Luft- und Raumfahrt (DLR) PRIMARILY CAPABILITIES OF TT:

Development of functional layers for energy, aircraft and space applications (like fuel cells or modules for space crafts) by coating technology

#### **MAIN GOALS OF TT IN DESHNAF:**

Eliciting the prospects of thermal plasma technology in the field of DESHNAF by

- Compiling of different plasma systems for the deposition of nanocomposite super-hard films
- Comparing the behaviour of various material substrates during plasma deposition
- Comparing of DLR's expertise to that of project partner's technologies
- Finding of new projects and partners for this technology

#### TASKS OF DLR IN CA DESHNAF

#### **Organisation and Performance:**

Workshop (20-22 September 2004, Stuttgart):

"Comparison of different plasma systems for the deposition of nano-composite super-hard films and the behaviour of various material substrates during plasma deposition"

#### **Identification:**

Figuring out the possibilities of thermal plasma technology in the field of DESHNAF

#### **Participation:**

On all DESHNAF workshops and meetings, dissemination of know-how in conferences (Eilat, St. Petersburg)

#### **Testing:**

Coating of B4C as high-temperature- and oxidation-resistant layer on reinforced SiC fibres with SiC matrix (SiC/SiC) gave encouraging results but no super-hard films

#### LIMITATIONS AND CHANGES DURING THE PROJECT

#### **Lessons learned from Plasma workshop:**

Plasma spraying is a challenge for producing ultra-hard coatings (no real CVD or PVD process)

- Powdery feedstock/suspensions do not yield nano-composite coatings as there is no complete evaporation phase
- It can be done on a laboratory scale but does not yet have meaning under production related considerations

 Only little cooperation of DLR with other project partners (PhD project with Uni. Surrey and Nigeria failed, STREP proposal "Plastofil" declined by EU commission)

#### **Important alterations at DLR in 2006:**

- Two competent know-how disseminating colleagues left DLR at the end of 2005
- Awarding "Centre of Excellence" (cooperation of two DLR institutes specialized on coating processes) offers new chances

#### **PERSPECTIVES**

## (I) SURFACE – Centre of Excellence

# **Contributions:**

- oxidation protective coatings
- graded and other advanced microstructures
- multilayered soft/hard- coatings and their behavior at high temperature
- erosion resistant coatings
- thermal protective coatings

#### Methods available now at DLR to develop and manufacture coatings:

- Multi-source and plasma- assisted magnetron sputtering
- Numerous RF- and DC- plasma spraying techniques
- High-speed PVD
- Electron-beam physical vapor deposition

# (II) What DLR can contribute in carried on DESHNAF

#### **Evaluation of coatings and processes:**

- Plasma diagnostics during PVD and PS processing
- Measurements of coating properties (adhesion, uniformity, thickness, composition, etc.)
- Microstructure characterization
- Cyclic oxidation test rigs
- Thermo-mechanical testing at high temperature including temperature gradients
- Interaction of coatings with metallic (Ni-base, Ti-base) and ceramic substrates

#### (III) Why DESHNAF should be continued

#### From a technological point of view:

 Thermal spraying of nano-composites needs a real vapour deposition process (TPCVD, Plasma Flash Evaporation - PFE). Investigations with liquid or gaseous precursors are required.

#### From a strategic point of view:

 The strength/fitness/skill of an existing consortium developed in three years consisting of excellent partners in R&D and industry should be used.

#### From a perspective point of view:

 DESHNAF's industrial relevance can be increased by a more comprehensive definition like compositional materials/multi layers at elevated temperatures for super-hard or corrosion/oxidation resistant applications.

#### (IV) High temperature applications

Promising materials for high thermal protection:

- Thin plates made of sintered SiC for plate-tin heat exchangers in coal or biomass power plants
- Carbon fibre reinforced silicon carbide (CMC = ceramic matrix components) for brake discs or racks for high-temperature processes (annealing)

#### Restriction:

Low stability against oxidation/corrosion at higher temperatures

#### Potential goal:

Development of oxidation protective/ environmental barrier coatings on SiC or CMC designed for <u>elevated temperatures</u> (> 1500 °C)

# Tel Aviv University

The main task of TAU in DESHNAF was to organize the INTERNATIONAL CONFERENCE ON SUPERHARD COATINGS. The conference took place in Ein-Gedi, Israel, on 27 Feb.-2 March 2006

The local Organizing Committee was comprised of:

- Members of the DESHNAF consortium (4)
- TAU students (9)
- TAU staff (5)
- Staff from other Israeli organizations (4)
- Spouses (4)

The students assumed major responsibilities and did a great job!

#### **Publicity**

- Direct e-mail to select targeted group identified via literature and web search
- Fliers and posters at related conference
- Announcements in publications
- Web
- Logo design contest
- No snail mail, nor shot-gun publicity

#### **Participants**

- 94 registered participants from 23 Countries
- Biggest delegations from Israel, Germany, Russia, UK, USA, and Poland
- Over 30 accompanying persons

#### Technical Program

The Program Committee included the following members:

Last Name	First Name	Institution	Task
Avni	Rudi	ECIL	Program Chairman
Boxman	Ray	TAU	Chairman
Zhitomirsky	Vladimir	TAU	Editor
Raveh	Avi	BGU	Editor
Shashurin	Alexey	TAU	Conference Secretary
Grach	Dmitry	TAU	Assistant Editor

The technical program included 55 Papers in six sessions:

- (1) Techniques for depositing superhard and nano-structured coatings
- (2) Characterization techniques for superhard and nano-structured coatings
- (3) Interfaces impurities and contaminants in the substrate / coatings region
- (4) Thermal stability and properties of superhard and nano-structured coatings
- (5) Tribology and mechanical properties of superhard and nano-structured coatings
- (6) Industrial applications of superhard and nano-structured coatings.

# Social Program and Atmosphere

- Guiding Principles:
  - The most important sessions of the conference are the coffee breaks
  - ➤ The most important "business" of the conference is to engender informal contact between the participants
- Setting Kibbutz Ein Gedi
  - > Oasis in Judean Desert
  - Overlooking Dead Sea and Nahal Arugot nature reserve lots of natural beauty
  - > Isolated 30 min to the nearest settlement
  - > All meals in Guest House dining hall
  - Pleasant lawns, botanical gardens, for informal contacts
  - > Excellent auditorium
- Social Events
  - > optional pre-conference tour of Jerusalem
  - > welcome reception
  - hike in the adjacent nature reserve
  - spa party
  - participants' lounge

## in vino veritas ad libra

- walking tour of Kibbutz Ein Gedi and its botanical garden
- > tour to Masada's historical/archeological site
- conference banquet
- accompanying persons' tour of Qumran Site of Essene community, Dead Sea scrolls

#### Special Issue in Surface and Coatings Technology

Editors: Vladimir Zhitomirsky and Avi Raveh

34 papers submitted, 21 published

Expected publication date Feb/March 2007

#### Dissemination

- SVC Bulletin article (aimed at coating practitioners) is in preparation, with Vladimir, Avi, Stan
- Invited talks, seminars, by Boxman
- French Electrical Arc Society
- ION 2006 (PL)
- Charles University, (Prague)
- Thin Films 2006 (Singapore)

#### **Exchange of Personnel**

- TAU Graduate student Dimitry Gindin, at Surrey University.
- Boxman at DESHNAF parters:
  - ➤ The Institute for Chemistry of Inorganic Materials at the Technical University of Munich, and the Materials Research Division
  - > Max Planck Institute for Plasma Physics.
- Boxman at other plasma & materials laboratories
  - University of Limoges
  - > University of Uhzgorod
  - > Hungarian Academy of Science
  - Charles University

#### Collaboration & Proposals

- Avi Raveh (BGU) on oxide coatings (IL gov't)
- Marcus Schwarz, Edwin Kroke (U. Freiberg) nanorod reinforced coatings (Kurt Lion Fund)
- R. Adrievski (Moscow) proposal to Russian-IL fund
- Discussions on various EU-FP7 proposals

# University of Sheffield

## **Summary of Activities**

- Participation in 4 DESHNAF workshops, and HARDCOAT conference
- 12 Presentations at DESHNAF workshops and 1 presentation at HARDCOAT conference
- 2 exchange visits to DESHNAF partners
- 2 exchange visits to EU institutions outside DESHNAF.
- Dissemination of nanocomposite coating results at 14 international conferences including 7 in Europe
- 20 journal papers plus 1 book chapter published
- Involved in 2 STREP project proposals (RIHPEN and PLASDOFIL)

#### **Exchange Visits**

#### The following institutions sent visitors to Sheffield

- University of Surrey (DESHNAF partner)
- Technical University of Munich (DESHNAF partner)
- University of Cyprus

Moscow State Institute of Steel and Alloys

#### Sheffield visited the following institutions

- Surrey University, and other DESHNAF partners such as DLR during DESHNAF workshops
- Dissemination and information exchange visits also made to organisations outside DESHNAF, such as VTT Technical Research Center (Finland), Moscow State Institute of Steel and Alloys (Russia), Groningen University (Netherlands) (no charge to DESHNAF)

## Dissemination within DESHNAF and within Europe

Sheffield participated in all DESHNAF workshops and the HARDCOAT conference. Dr Adrian Leyland, Kostas Kanakis and John Eichler attended the latter event and Dr. Leyland presented an Invited paper as well as chairing a session. Sheffield also organised the successful Tribology workshop in Sheffield (11-13th January 2006), which included participation from all partners as well as from Groningen University and the University of Cyprus. Further details of this event are available on the DESHNAF web site.

In addition Sheffield researchers presented at the following European conferences:

#### IOMMM Materials Congress 2004 (London)

Prof. Matthews presented a talk on Nanocomposite Coatings.

## Congress of the Spanish Materials Society 2004 (Valencia, Spain)

Prof. Matthews presented the opening Keynote invited lecture on nanocomposite tribological coatings.

## IoP Plasmas, Surfaces and Thin Films Conference 2005 (London)

Prof Matthews' talk was entitled "Developments in Plasma-Based Processes for Production of Wear Resistant Surfaces".

# EMAG - NANO 05 Imaging, Analysis and Fabrication on the Nanoscale (Leeds)

Martynas Audronis, presented a poster entitled: "A TEM study of the structure of magnetron sputtered chromium diboride coatings" at this meeting.

#### PSE2006 (Garmisch-Partenkirchen, Germany)

Sheffield presented 10 papers in either poster or oral form.

#### **Dissemination Outside DESHNAF**

## SVC Techcon2005 (Washington, USA)

Prof Matthews chaired two sessions. He also presented the opening Invited Presentation at the 17th Annual Workshop of the Advanced Coating and Surface Engineering Laboratory (ACSEL) of the Colorado School of Mines. The presentation was entitled "Enhanced Tribological Performance through Plasmabased Processes".

#### ICMCTF2006 (San Diego, USA)

The Sheffield team participated in the largest number of papers (15) of any research group (spread across 4 of the Symposia). For the first time there were more participants from outside the USA than from within that country, with a major European presence.

#### ISEC 2006 (Seattle, USA)

Prof Matthews presented the Opening Plenary Paper entitled "The role of nanocomposite coatings in surface engineering".

#### MS&T2006 (Cincinnati, USA)

Prof Matthews gave an Invited Keynote Paper (co-authored with Dr Adrian Leyland and Prof Kenneth Holmberg, VTT, Finland) entitled "Nanocomposite Coatings for Enhanced Wear Resistance".

At each of the above events it was possible to hold discussions with researchers working on nanocomposite coatings from all over the world (including DESHNAF partners in many cases).

# **EU Proposals**

# Sheffield participated in 2 STREP proposals:

- RIHPEN (Role of interfaces in high performance nanocomposites) involving 9 partners, including 3 from DESHNAF
- PLASDOFIL (Plasma deposition of high dielectric oxide thin films for capacitors, microwave filters and sensors) – involving 8 partners, including 4 from DESHNAF

Sheffield is now actively involved in discussing revised and updated versions of these projects with DESHNAF (and other) partners. In addition, following discussion between partners (including at the last workshop in Prague), we are involved in the development of potential FP7 projects in topic areas such as medical devices and other EU priority themes.

## **Concluding Remarks**

- The project has facilitated the formation and enhancement of many new contacts, including DESHNAF partners and also other collaborators such as Groningen University, University of Cyprus and Moscow State Institute as well as companies in new member states such as the Czech Republic.
- DESHNAF has facilitated exchanges and discussions on exciting new EU projects and research collaborations.
- DESHNAF has provided opportunities to meet researchers active in nanocomposite coatings from all over the world including less developed countries.
- The DESHNAF workshops and conference have been particularly effective forums for meeting researchers from Academia and Industry and ensuring that our research is pursued more effectively.

# Max Planck Institute für Plasmaphysik

H. Maier, D. Levchuk, F. Koch, B. Cieciwa, Th. Köck, H. Bolt

Max-Planck-Institut für Plasmaphysik,

Euratom Association, D-85748 Garching

Materials Research Division

#### The Institute

Dedicated to the development of **Nuclear Fusion** into an energy source by magnetic confinement of high temperature plasmas

The Division was founded in 1999, with the main tasks of:

- Surface processes on plasma-facing materials in laboratory and fusion devices
- Hydrogen retention and permeation / diffusion barriers
- Thin films and materials synthesis
- Fusion device components development (also materials)

Coating Activities of the Materials Research Division:

 Development of oxidation-resistant tungsten coatings for application in future fusion reactors

- Sputter deposition of tungsten based coatings and subsequent oxidation investigation
- Diffusion barriers: ceramic thin films by sputtering and vacuum arc

Control of crystal structure by control of deposition parameters

#### **IPP's activities withinDESHNAF**

#### Workpackage 3

With this background IPP performed the literature study on the influence of plasma variables on the coatings properties. The results were presented at the first DESHNAF workshop (i.e. workpackage 2) and at the International Conference. The title of the paper is "The influence of deposition variables on mechanical properties of coatings" by Denis Levchuk

<u>Workpackage 4</u> Workshop on the methodology for correct measurement of hardness at the Technical University Munich

IPP benefited from this workshop especially through our staff gaining expertise in nano-indentation measurements

<u>Workpackage 5</u> Workshop on corrosion resistance and thermal stability, Eilat IPP presented work on development of oxidation-resistant W-based alloys by sputter-deposition

Valuable contact made, hopefully more exchange in the future, especially with Prof. A. Cavaleiro, University of Coimbra, Portugal

#### Workpackage 8

Contact established with Prof. Jochen Schneider, RWTH Aachen, Germany Workpackage 10 Seminar on Industrial Applications, Prague

IPP was approached by several industrial contacts.

Visit by Oerlikon Balzers in Garching was arranged for Dec. 06 with special Interest in alumina

#### **Summary**

- IPP distinctly profited from several workpackages of DESHNAF by gaining of knowledge and skills
- Several potentially valuable scientific contacts were made
- Several potentially interesting contacts with industry were made (reaching beyond Europe)
- Contacts made will be valuable for IPP beyond the period of DESHNAF

# **University of Surrey**

The Surface Analytical Laboratory

School of Engineering, University of Surrey, Guildford, Surrey, UK

# **Summary of Activities**

- Participation in 3 DESHNAF workshops and the HARDCOAT conference
- 1 presentation at DESHNAF workshop, 2 presentations at HARDCOAT
- Participation in 6 exchange visits (4 visitors to Surrey, 2 from Surrey to hosts)
- 2 exchange visits involved EU institutions outside DESHNAF
- Dissemination of superhard and nanostructured coating results at 3
   European and 2 international conferences

 7 journal papers published on superhard and nanostructured coatings in 2005-2006

- Mailshot to over 500 eastern and western European scientists involved in coatings R&D
- Involvement in NMP STREP project proposal (RIHPEN)

## **Exchange Visits**

#### The following institutions sent visitors to Surrey:

- Max Plank Institute, Stuttgart, Germany (DESHNAF partner)
- Tel-Aviv University, Israel (DESHNAF partner)
- University of Sheffield, UK (DESHNAF partner)
- University of Cyprus, Cyprus
- Ben Gurion University, Israel

# Surrey visited the following institutions:

- Tel-Aviv University, Israel (DESHNAF partner)
- ENEA research centre, Brindisi, Italy

#### **Dissemination outside DESHNAF**

- International Conference on Metallic and Ceramic Thin Films (ICMCTF), San Diego, USA (2005/2006)
- European Conference on Applied Surface and Interface Analysis (ECASIA),
   Vienna, Austria (2005)
- European Congress on Advanced Materials and Processes (EUROMAT),
   Prague, Czech Republic (2005)
- Novel Applications of Surface Modification, Chester, UK (2005)

#### **EU** proposals

STREP submission to FP6 NMP programme made in Sept. 2005:

A consortium of 9 partners submitted a proposal entitled:

'Role of Interfaces in High Performance Nanocomposites' (RIHPEN)

#### **Concluding Remarks**

- 1. Technical reports very useful review documents
- 2. The workshops provided an excellent platform for formal presentation and informal discussions on important topics for the future development of superhard and nanostructured coatings
- 3. HARDCOAT very successful focussed international conference in an environment conducive to discussion
- 4. Exchanges involving both DESHNAF and other EU institutions have lead to strengthened and new collaborations
- Useful collaborations made with DESHNAF partners resulting in new EU STREP proposal

# Ben Gurion University of the Negev

#### Coating Technology available at BGU

- 1. Dual Frequency technology (Microwave/Radio-Frequency) MASDAF
- 2. Inductive RF Plasma
- 3. Magnetron RF or DC Sputtering

- 4. Plasma-Enhanced Chemical Vapor Deposition (100-450°C), (PECVD)
- 5. Chemical Vapor Deposition (CVD) and Metal-Organic CVD (MOCVD)
- 6. Electron Beam Evaporation

#### **BGU Activities**

- Literature Study and Review: Thermal Stability of Superhard Coatings (together with ECIL)
- Organization of DESHNAF Workshop # 3: Thermal Stability and Wear Sept. 21-23, 2005, Eilat, Israel
- Assistance in organization of the Inter. Conf. on Superhard Coatings, (organized by TAU) 27 Feb.27 March 2, 2006, Ein Gedi, Israel: membership of the organizing and program committees, co-editor of the special issue of Surface and Coating Technology.
- Participation in 4 DESHNAF workshops and in the Hardcoat Conference

### **Literature Study and Review**

Study: Thermal Stability of Superhard Coatings

- Review of the literature as well as private knowledge on superhard coatings: Monolayers, Superlattice and Nanocomposite coatings.
- The study focused on various superhard coatings: compositions, miscible and immiscible superlattices, and nc-MeN/nitride superhard coatings.

# **DESHNAF Workshop # 3** Thermal Stability and Wear Sept. 21-23, 2005 **Subjects:**

- Thermal stability and stresses up to ~1200°C as a function of nanocrystallite sizes =10 nm and substrate materials
- Oxidation resistance as a function of the deposition temperature and nanocrystallite size
- The role of interfaces between substrate materials and deposited films or multi-layer films to increase thermal stability and oxidation resistance
- Wear resistance of hard and superhard coatings

## **Contribution to DESHNAF Workshops:**

- WS #1: Comparison between Plasma Deposition Techniques (20-22 September 2004):
  - The advantages of the Inductive Radio-frequency Plasma Technique in surface modifications, and the production techniques of Micro-Composite Material
  - > Behavior of metal substrates in a PACVD system during hard coatings deposition
- WS #2: Hardness Measurements (Nov. 9-11, 2004):
  - Hardness, Fatigue and Erosion Resistance of Plasma Treated Ti Alloy
- WS #3: Thermal Stability and Wear (Sep. 21-23, 2005):
  - Phase diagrams of nanoparticles
- WS #4: Tribology and Wear (Jan. 11-13, 2006):
  - A microstructurally based method for the evaluation of the stressstrain relations in surface layers of metals under frictional contact

- International conference on superhard coatings,
  - ➤ Thermal Stability of Nanostructured Superhard Coatings
  - Internal Stress of TiAl(B)N at High Temperatures
  - > Tribological Properties of Duplex Treated TiN/TiCN Coatings on Plasma Nitrided PH15-5 Steel

# 5 papers were published during the years 2004-2006

## Dissemination of Knowledge Outside of DESHNAF

- Inter. Conf. on Metallurgical Coatings and Thin Films (ICMCTF), San Diego, USA, 2005
- Inter. Conf. on High Technology Plasma Processes (9HTTP), Saint Petersburg, Russia, 2006
- 11 other lectures in BGU, TAU, Holon and Technion-Haifa, Israel (workshopes and seminars)

## **Exchange of Visits BGU visited the following universities:**

- Surrey University, UK
- Technical University of Munich (TUM), Germany

#### BGU accepted visitors form the following universities:

- University of Lodz, Poland
- University of Montreal, Canada

### **Summary and Conclusion:**

- Discussion of results and exchange of knowledge with DESHNAF partners
- Formation of new contacts with several researchers from DESHNAF partners and with other colleagues from Europe (Portugal, Cyprus, UK, Switzerland and Germany)
- Meeting with non-DESHNAF colleagues from Poland, Russia, Czechoslovakia, Taiwan, Mexico, Australia, India, and Japan
- Formation of new contacts with researchers for the purpose of collaboration and design of new EU STREP proposals