from Tsukuba, Japan to the world

NIMS NOW International

National Institute for Materials Science

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Special Features

Nanotechnology/Materials Research Supporting the Creation of a Sustainable Society III

In-Situ Observation of Nanoindentation in TEM

—Direct Observation of Plastic Deformation in Local Regions—

Takahito Ohmura, Kaneaki Tsuzaki
Physical Metallurgy Group
Steel Research Center (SRC)

Plastic deformation of materials at the macro level is the result of combined local deformations. Many materials have an inhomogeneous nanoscale structure containing grain boundaries, precipitates, and other discontinuities which have important effects on local deformation behavior. Clarification of the mechanism of local behavior will make it possible to design microstructures capable of controlling of these factors, and thus will contribute to improved properties in actual parts and structural components.

As a method of analyzing deformation behavior in local regions, the authors applied a technique called nanoindentation. Nanoindentation is a new technique in which a pyramidal probe is forced into the material surface with a micro-load of μ N level (1/10,000 gf), and the depth of the indentation caused by the probe is measured. The size of the indentation formed on the material surface is on the order of 10-100 nm, and it is possible to measure the hardness of internal parts of individual grains and single precipitates. We are also developing a more advanced version of this technique, in which nanoindentation is performed in a transmission electron microscope (TEM), enabling direct *in-situ* observation of the deformation behavior of the material. The elementary process of plastic deformation is understood as a behavior in which lattice defects called dislocations move within the material. Dramatic progress in clarifying the mechanism of local deformation can be expected by applying this combination of nanoindentation and TEM to the investigation of dislocation behavior.

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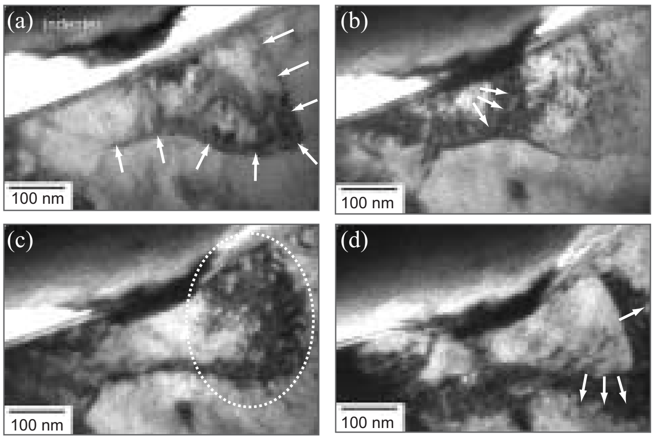


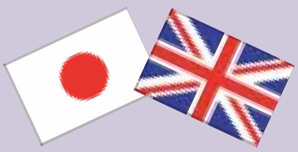
Fig. Example of *in-situ* observation of nanoindentation in a TEM. The material is martensitic steel.

in this issue

NIMS News

NIMS Students Went to Cambridge

In front of the Cambridge Nanoscience Centre. Prof. Mark Welland, Director of the IRC, is in the center.



Research Presentation

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Casting Process for Reducing the Size of Inclusions to Nanoscale in Steel

—Utilization of Impurities in Steel by Rapid Cooling Process—

Yoshinao Kobayashi, Kotobu Nagai
Metallurgical Processing Group
Steel Research Center (SRC)

Nonmetallic compounds which are formed or introduced into steel during the steelmaking process are called inclusions. Conventionally, efforts are made to remove these inclusions since they deteriorate the quality of the product. However, it is possible to minimize these adverse effects and actually improve the mechanical properties of steel products by reducing inclusions to nanosize.

The Metallurgical Processing Group is involved in research aimed at controlling the precipitation of inclusions, and suppressing inclusion growth and refining inclusions, by increasing the cooling rate in the cooling process during steel casting. Steel is normally cast using a water-cooled copper mold. The cooling rate is related to the mold width, in that cooling can be accelerated by adopting a narrower mold width. In our present research, we used a technology called strip casting, in which molten steel is solidified continuously into a thin strip form as it flows between water-cooled copper rolls. Strip casting is particularly useful in this research, as it has the highest cooling rate among current steel casting processes. In the present work, we conducted experiments on materials with composition similar to scrap steel containing copper, sulfur, phosphorus, etc. from the viewpoint of utilization of steel scrap (see **Fig. 1**).

As a result, it was found that compounds of unprecedentedly small size (average size: approx. 15 nm) precipitate as inclusions in the cast steel. Analysis revealed that the main component of these inclusions is copper sulfide ($Cu_{2-x}S$; **Fig. 2**). This process increases the strength of the steel by about 1.5 times in comparison with the conventional material (SS400 standard), and also improves the strength-ductility balance (**Fig. 3**). To identify the cause of this strength increase, the strength properties of the as-cast steel and the same material after annealing (**Fig. 4**) were investigated. The results of this analysis showed that the existence of nano-inclusions makes a larger contribution to strength improvement than the difference in the refinement of the matrix structure (**Fig. 5**).

Precipitation of MnS, which is a coarse inclusion, is a problem in the conventional casting process. However, as an advantage of strip casting, because this is a rapid cooling process, precipitation of MnS is suppressed, resulting in precipitation of $Cu_{2-x}S$, and the presence of coexistent impurity phosphorus expands the temperature range for easy precipitation of $Cu_{2-x}S$.

The Metallurgical Processing Group is now expanding its research on the formation and use of nano-inclusions based on the results described above.

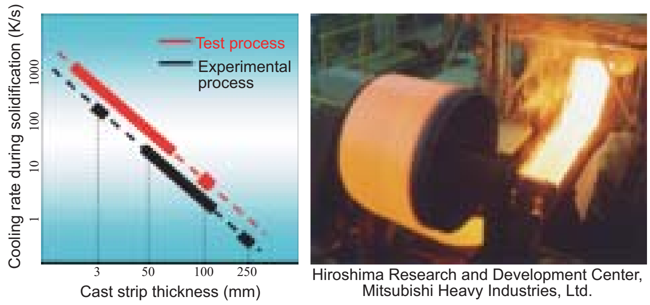

Fig. 1 Relationship between casting thickness and cooling rate, and illustration of strip casting method.

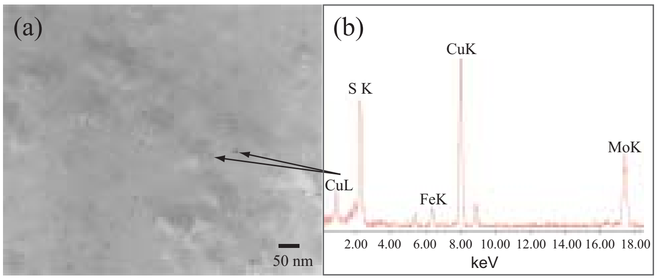

Fig. 2 Micrograph and composition analysis of formed inclusions.

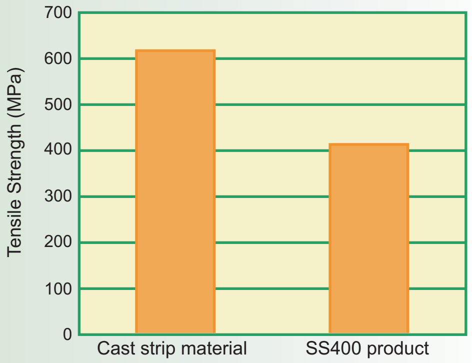

Fig. 3 Example of strength improvement of cast strip.

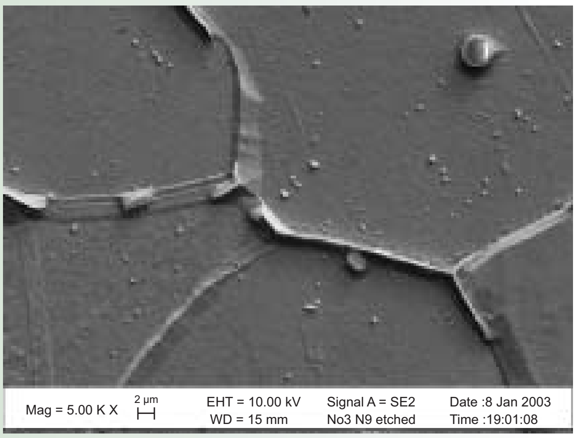

Fig. 4 SEM image of inclusions in strip-caster product after annealing.

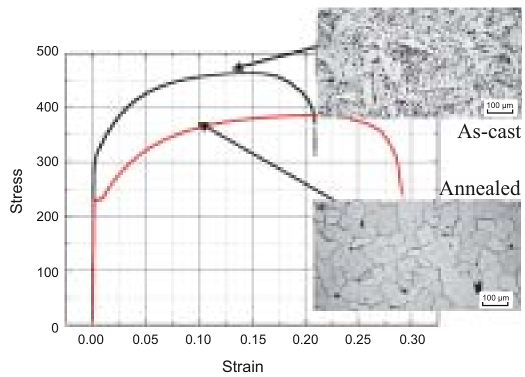

Fig. 5 Comparison of strength properties of as-cast strip material and annealed material.

For more details: http://www.nims.go.jp/mpg/index_e.html

Hydrogen Trapping of Nanoparticles

Fu-Gao Wei, Kaneaki Tsuzaki
Physical Metallurgy Group
Steel Research Center (SRC)

Hydrogen is the smallest of all elements, and is absorbed/desorbed by iron and steel at room temperature. Generally, the amount of hydrogen in iron and steel is extremely small, being several wt. ppm or less. However, hydrogen accelerates brittle fracture of materials, which is called hydrogen embrittlement, because hydrogen tends to accumulate at the stress concentration region ahead of a crack tip. Moreover, susceptibility to the hydrogen embrittlement increases with material strength. For this reason, susceptibility to hydrogen embrittlement is one of the properties which are evaluated most strictly in high strength steels.

As an approach to reducing the susceptibility to hydrogen embrittlement, introduction of fine particles which interact strongly with hydrogen into the material is considered to be able to trap the hydrogen and prevents it from migrating to the crack tip. We have found that titanium carbides (TiC) and vanadium carbides (VC) precipitates in steels, which have been known as precipitation-hardening particles, are of such a function of hydrogen trapping. We also found that hydrogen can be trapped most effectively if the sizes of precipitate particles are reduced to nanoscale.

Fig. 1 and **Fig. 2** are lattice images of nanosized TiC particles that precipitated on the (100) plane in iron. These TiC particles take a shape of disk. Although it has been considered extremely difficult until now, we have been successful in application of high resolution transmission electron microscopy to observation of the microstructures of low alloy steels and made it possible to quantitatively evaluate the crystallographic structures of the nanoparticles and their relation to the hydrogen trapping property. According to an image analysis of **Fig. 2**, the TiC nanoparticle was known to form a semi-coherent interface with the Fe matrix, as schematically illustrated in **Fig. 3**. On the interface, there are distorted regions that act as hydrogen trapping sites. These sites include the misfit dislocation core (indicated by the letter T), the coherent interfacial area adjacent to the core, and the elastic strain field neighboring this interface. These sites are different in the intensity of interaction with hydrogen. The interaction increases approaching the dislocation core.

In the future, we plan to develop ultra-high strength steels with excellent resistance to hydrogen embrittlement utilizing the hydrogen trapping function of TiC and other nanoparticles.

For more details: http://www.nims.go.jp/mpg/index_e.html

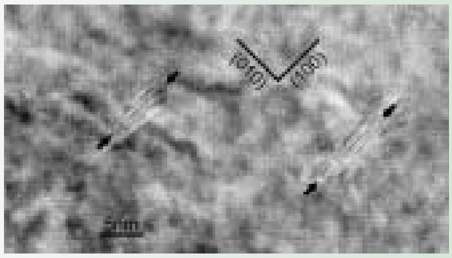

Fig. 1 High resolution image of TiC nanoparticles precipitated on the (100) plane in Fe.

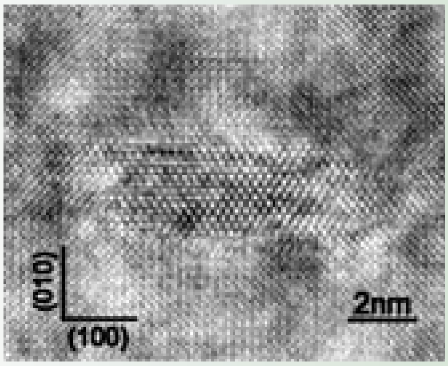

Fig. 2 High resolution image of a TiC nanoparticle precipitated in Fe.

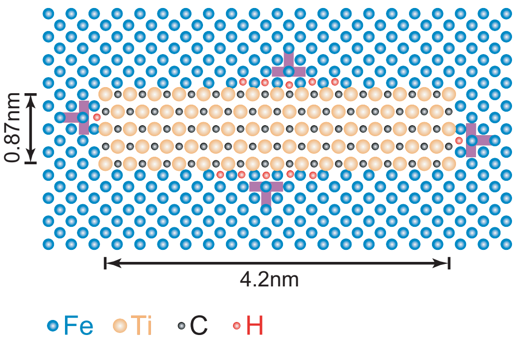

Fig. 3 Schematic diagram of hydrogen trap site at interface between TiC nanoparticle and Fe.

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In-situ Observation of Nanoindentation in TEM

The **accompanying figure** shows one example of observation results. The deformation process is recorded on video. The **figure** shows characteristic frames taken from one such video. The specimen is martensitic steel, and a grain boundary is observed (shown by arrow in **(a)**). The probe can be seen in the upper left part of the **figure**, and is arranged to press into the specimen in the lower right. In **(b)**, a dislocation which is migrating from the area of contact between the probe and the material toward a grain boundary can be observed. Under further pressure, the dislocation stops temporarily near the grain boundary (as shown in **(c)**), and then simultaneously advances into the adjacent grain **(d)**. The fact that migration of the dislocation is impeded in the vicinity of the grain boundary clearly indicates that grain boundaries offer large resistance to deformation. In particular, this is extremely important knowledge for understanding the deformation mechanism of polycrystals. (These results were obtained in joint research with Prof. J. W. Morris of the University of California at Berkeley.)

For more details: http://www.nims.go.jp/pmg/index_E.html

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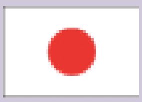
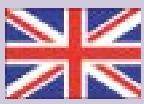


NIMS Students Went to Cambridge



(July 11-15, Cambridge) -- The 2nd NIMS-IRC Nanotechnology Summer School was held at Cambridge University in the UK. The IRC is an interdisciplinary research collaboration group in nanotechnology established by three British universities, Cambridge, London, and Bristol. The participants from the UK were 18 students who are involved in research at the IRC and from NIMS, 17 students conducting research at NIMS. The sessions included presentations on recent research achievements and active discussions. Considerable progress since last year could be seen in both the scientific aspect and in presentation techniques. In addition to the summer school sessions, the participating students also deepened their friendship through sports such as bowling, soccer, and punting. The 3rd Summer School is scheduled to be held at NIMS next summer.

High Temperature Thermal Stabilization of Microstructure using Nano-Nitride Control

Masaki Taneike
Mitsubishi Heavy Industries, Ltd.
(Former Researcher at the Steel Research Center)

Kota Sawada
Creep Group
Materials Information Technology Station (MITS)

Fujio Abe
Heat Resistant Design Group
Steel Research Center (SRC)

In thermal power generation, high temperature, high pressure steam is used to drive a turbine- generator, producing electric power. Use of higher steam temperature/pressure improves generating efficiency, reducing fuel consumption, which in turn reduces CO_2 emissions. For this, high strength heat-resistant steel (steel for high temperature service) which is structurally stable at elevated temperatures is necessary. Dispersion of particles in the material matrix is one effective method of strengthening heat-resistant steels, but if the particles agglomerate due to diffusion during high temperature use, the spacing between particles will increase, resulting in reduced strength. In order to develop heat-resistant steels with excellent high temperature strength, the Heat Resistant Design Group is searching for nanosized particles which are thermally stable and thus do not coarsen in high temperature use.

Ferritic heat-resistant steels for use in thermal power plants usually contain a maximum of 12 % chrome for resistance to high temperature oxidation and small amounts of alloying elements such as tungsten, vanadium, niobium, carbon, nitrogen, etc. for improved high temperature strength. Particles of carbides and nitrides (compounds of metallic atoms and carbon or nitrogen, respectively) are useful in strengthening, but chromium carbides are prone to coarsening. In contrast, vanadium and niobium nitrides are nanosized and have the property of resisting coarsening. The main type of particle used in conventional steel is chromium carbide, but because these carbides coarsen when used under high temperature conditions, there are limits to the high temperature strength which can be achieved using this method.

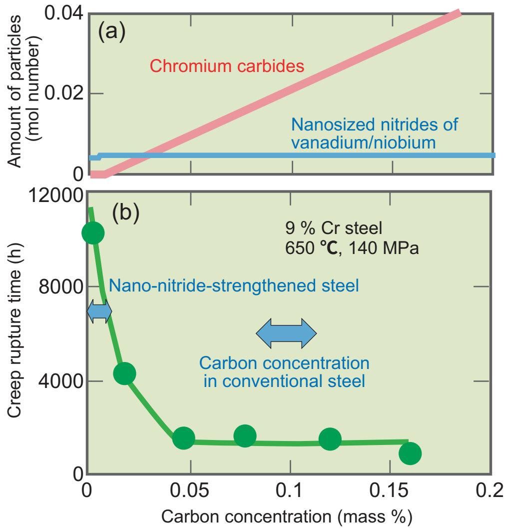


Fig. 1 Carbon concentration dependence of creep rupture time of 9 % Cr heat-resistant steel at 650 ℃ and 140 MPa. In the figure, (a) shows the types of particles after heat-treatment and the calculated values of their amounts.

In the present research, the amounts of carbides and nitrides in 9 % Cr heat-resistant steel (nitrogen: 0.05 %) were calculated using a thermodynamic program, Thermo-calc, revealing that the contents of vanadium and niobium nitrides become larger than that of chromium carbides when the carbon concentration is reduced to 0.02 % or less (**Fig. 1(a)**). Therefore, specimens with different carbon concentrations were prepared, and a 650 ℃ creep test was performed (measurement of deformation rate and time to rupture under constant temperature and load conditions). The results showed that the material resists deformation and the creep rupture time (rupture strength) is greatly improved by dispersion of only nanosized vanadium and niobium nitrides (**Fig. 1(b)**). **Figure 2** shows electron microscope images of the grain boundary after heat treatment. In the conventional steel, chromium carbides 100-300 nm in size are distributed in this region, but these undergo further coarsening during the creep test. In the nanonitride-strengthened steel, the nanosized nitrides are finely distributed and are stable even at high temperature.

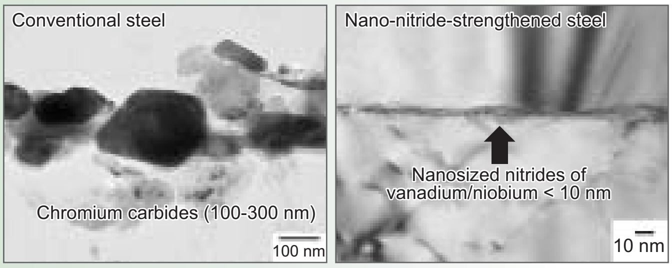


Fig. 2 Chromium carbides in conventional steel, and dispersion of nanosized nitrides in ultra-low carbon steel developed in this research.

For more details: http://www.nims.go.jp/creep/creep_e.html
http://www.nims.go.jp/hrdg/english/0framepage.html

NIMS News

Visit by South African Minister of Science and Technology

Minister Mangena (second from left) with other delegation members and President Kishi (center).

(June 3, NIMS) -- Mr. Mangena, Minister of Science and Technology of the Republic of South Africa, and Dr. Ngubane, South African Ambassador to Japan, visited NIMS. Since NIMS reached an agreement for joint research on superalloys with two research institutes in South Africa and exchanged MOUs with its counterparts in May of last year, the two sides have actively conducted exchanges of personnel and joint research. During this visit, the South African guests received a general explanation of NIMS, which was followed by a discussion of future research cooperation with President Kishi. The visitors then observed laboratories engaged in research on high temperature materials and biomaterials.

Science Camp 2005 for High School Students

(July 27-29, NIMS) -- Science Camp 2005 was held at NIMS and a total of 15 high school students from across Japan visited NIMS during the event. The NIMS researchers and participating students were soon on friendly terms and engaged in lively practical training, in which the students conducted a variety of experiments involving metals. The students actively questioned the researchers and enjoyed experiments that most high-schoolers can never experience. These exchanges with NIMS researchers engaged in leading-edge research appeared to fire the students' interest in materials research.

Mini-Doctorate Course 2005 for Middle School Students

The scene during an experiment.

(August 3-5, NIMS) -- NIMS held the Mini-Doctorate Course 2005 for middle school students, which is under the auspices of the Ibaraki Prefecture Education Committee. Six middle school students from the prefecture visited NIMS for this event. The first day began with an orientation, followed by experiments with shape memory alloys, biomaterials, superconductors, and others. The second day featured various experiments with diamonds, and the third day provided the youngsters with a chance to make castings and an original key holder, giving the mini-Ph.D. students a rich and varied experience.

Tsukuba Express Train Starts in Service - Tsukuba-Akihabara in 45 Minutes -

New Tsukuba map.

(August 24, Tsukuba) - The long-waited Tsukuba Express (TX) train finally began its operation this summer. This line links Tsukuba where NIMS is located and Akihabara in central Tokyo in 45 minutes. Akihabara is Japan's discount electronics mecca and also gained fame as a center of subculture, such as gaming and animation recently. NIMS expects that the launch of TX will make your visit to NIMS a lot more convenient and exciting.

Appointment of New Vice President

(September 6, NIMS) -- Mr. Tetsushi Uehara, former Secretary General of Nuclear Safety Commission of Japan, was newly appointed as a Vice President of NIMS, replacing an outgoing Vice President, Dr. Hirose.

Mr. Testsushi Uehara

Biography

Completed Master's Course in Nuclear Engineering at the Engineering Research Dept. of the Tohoku University Graduate School, and joined the Radiation Section of the Atomic Energy Bureau, Science and Technology Agency (1974). Served as Senior Researcher at the No. 1 Research Group of the National Institute of Science and Technology Policy (NISTEP) and then worked for the Science and Technology Agency Director's Secretariat as Manager of the Public Relations Group, General Affairs Section (1989). Appointed as the Manager of the International Cooperation Group of the Japan Atomic Energy Research Institute (JAERI; 1991) and then as General Manager of the Business Planning Department, Japan Nuclear Cycle Development Institute (JNC; 1998). Became the Manager of the Accounting Section, Science and Technology Agency Director's Secretariat (1999). Named as the Deputy Director-General of the Minister's Secretariat Division, Cabinet Office (2002) and the Secretary General of the Nuclear Safety Commission of Japan, Cabinet Office (2004). Appointed as the Vice President of NIMS (2005).

■ International in Japan ■

Caterina Minelli (Italy)
ICYS Fellow (Jan. 2005 - Jan. 2006)
International Center for Young Scientists (ICYS)
Biomaterials Center (BMC)

[With ICYS Fellows, first from left, back row]

Konnichiwa! I'm Caterina Minelli, an Italian ICYS Fellow in NIMS. I come from beautiful Florence, where I studied physics before moving to Switzerland to complete my Ph.D. in Nanotechnology. I'm currently working at the International Center for Young Scientists (ICYS) in collaboration with Dr. Yamamoto of the Biomaterials Center (BMC) of NIMS. Together, we are interested in studying the interaction of nano-structured polymer surfaces with cells. How do cells interact with the environment? Do they have a kind of nose or a sense of touch to feel it? How and to what extent can we induce a certain behavior in them by modifying their surroundings? These are the kind of questions our research is addressing.

[With my colleagues in the lab, second from left]

Together with high level research, I'm experiencing in Japan a unique cultural enrichment, mostly due to the international environment I found in Tsukuba. Here you can have the chance of attending a wedding of people in kilt and in kimono, tasting Irish coffee made with Turkish coffee or celebrating the hanami (cherry-blossom viewing) with German sausages. In Tsukuba, and in NIMS in particular, people from different cultures and backgrounds become friends, and I think that this is what is needed today so that people will stop fighting each other around the world.

■ My Life Experiences in Japan's Science City, Tsukuba ■

Maje Phasha (South Africa)
Guest Ph.D. Student for Joint Research (Aug. 2005 - Dec. 2005)
High Temperature Materials Group
Materials Engineering Laboratory (MEL)

Multi-National Student/Research Assistant Members of HTM21 Project Group

Hello there! My name is Maje Phasha.

On my arrival in Japan, my initial concern was the heat. My quick adaptation to the Tsukuba climate made me realize that having come from the hot northern part of South Africa, Limpopo Province in particular, was actually a blessing in disguise.

Apart from the good environment provided by Tsukuba Science City, NIMS has created such a world-class working atmosphere that it makes me feel I am at home, even though far from home. Furthermore, the conditions here, reinforced by the friendly people, seem to have reduced the long lonely visit which I had originally feared, from July 2005 to December 2005, making it look very short.

Undoubtedly, I consider myself fortunate to be working in one of the most multicultural groups, the High Temperature Materials Group of the Materials Engineering Laboratory (MEL), since a common communication platform is promoted and established much more easily here.

I have rented myself a bicycle, which enables me to observe the nature incorporated in this city, which is beautifully covered mostly by green fields of rice. Being a sporting fanatic, both NIMS and Tsukuba have presented me with the opportunity to interact with a lot of people on a social basis, in which many interesting issues arise, in volleyball and soccer, respectively.

I am looking forward to my possible return to this wonderful city, mostly as part of my envisaged Ph.D. work, by participating in South Africa-Japan Joint Research and partly in fulfilment of my social explorations in Japan.

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National Institute for Materials Science

PUBLISHER
Dr. Masatoshi Nihei

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