Temperature Monitoring of an EM Environment

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Introduction

The ultimate performance of high resolution electron microscopes depends on a variety of factors including the stability of the temperature surrounding the instrument. Variations in temperature can cause drift of the specimen, the microscope electronics, and the mechanical tolerances in components such as microscope lenses and scan coils. For high resolution work or for image reconstruction that requires acquisition of multiple exposures over extended periods of time, it is critical that the temperature variations be strictly controlled. A typical manufacturer's specification for temperature stability of a room housing a transmission electron microscope (TEM) is a tolerance of <0.5°C/hour with fluctuations of <0.05°C/minute. While modern instrumentation rooms are carefully designed to maintain stable environmental conditions (O'Keefe *et al.*, 2004), it seems unlikely that these stringent specifications are routinely met in practice when the microscope is in operation and personnel are present in the room.

We have developed a temperature monitoring system for microscope rooms that is relatively inexpensive to install and very simple to operate. The system has been deployed in two instrument rooms housing TEMs and is used to continuously monitor the temperature at several locations within the room and on the instrument. The system has allowed us to precisely monitor temperature conditions during sensitive data acquisition sessions and begin to understand the influence of a variety of factors on the overall stability of the environment. The system is also capable of sending automated alerts and is useful in providing an early warning of instrument or environment malfunction.

Materials and Methods

The temperature monitoring system consists of a set of temperature sensors connected to modular data acquisition components that communicate to control software over a network. Most of the sensors consist of platinum resistance temperature detector (RTD) elements (Omega Instruments[1] or National Instruments[2]). RTDs are based on the principle that metal resistance increases with temperature and provide measurements with a precision of 0.3°C. These sensors operate within a range of -200 °C to 850 °C and are used to monitor the air, column and chilled water temperatures in the TEM room. In addition, we use another type of sensor to measure cryogenic temperatures in the liquid nitrogen cooled cold trap and specimen stage. Although RTDs can be used in subzero temperatures, the sensors are most reliably accurate between -100 °C and 100 °C, where the Callendar-Van Dusen Equation can be used to model the temperature vs. resistance to an accuracy of 1°C. Outside of this range, the accuracy of the RTDs decreases due to the complex thermal link between the sensing element and its entire environment and the effect of any measurement-induced self-heating of the sensor itself. For greater accuracy, an Omega CY7 cryogenic temperature silicon diode sensor, which reads fluctuations in voltage rather than in resistance, is used to measure the temperature of the liquid nitrogen cooled systems. An added benefit of these cryo-sensors is that they are able to withstand repeated cycling to low temperatures without mechanical failure. These sensors are accurate within 1% in the range of -173 °C to 32 °C and follow a standard temperature response curve that is approximately linear within the sensor range of -272°C and 202°C[3].

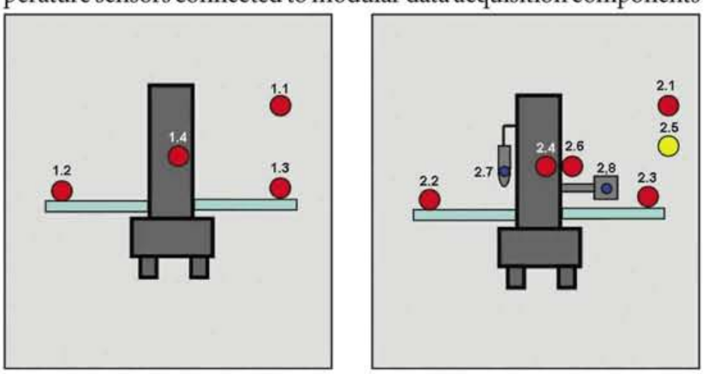

Figure 1: Layout of the temperature sensors in the two transmission electron microscope rooms. Room #1: sensor 1.1 measures ambient air temperature; sensor 1.2 measures temperature of incoming chilled water; sensor 1.3 measures temperature of outgoing chilled water; and sensor 1.4 in inserted into the column to a depth of 2". Room #2 has 4 additional sensors: sensor 2.5 is a second ambient air temperature sensor; sensor 2.6 is mounted on the outer surface of the TEM column; sensor 2.7 measures the temperature inside the LN_2 cold trap; and sensor 2.8 monitors the temperature of the cryogenic stage.

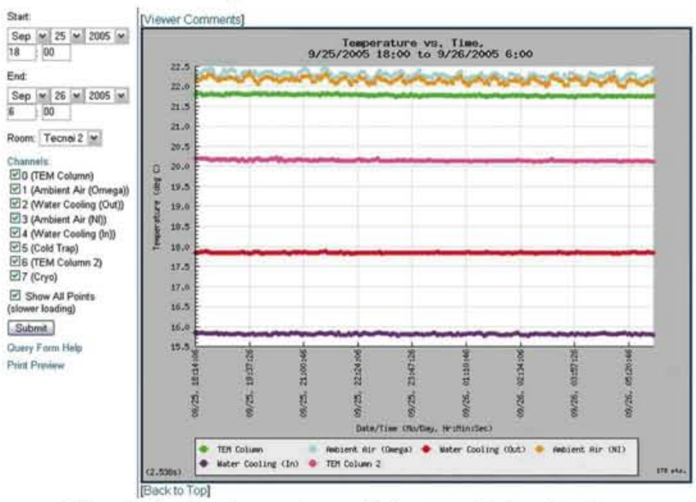

Figure 2: Baseline temperature profile for room #1 when the microscope was not in use. A web browser based interface reads the temperature data from the database and displays the results graphically.

The individual sensors are connected in a three-wire lead configuration to FieldPoint modules (National Instruments) using Teflon-insulated 26 AWG stranded nickel-plated copper lead wires of one-meter in length. With a separate voltage sensing lead to minimize the effects of lead resistance, the three-wire RTD configuration allows for more accurate measurements. Sensors were mounted using electrical tape with a layer of Omega thermally conductive silicone paste between the sensor and the surface of the equipment to be measured. The FieldPoint modules themselves are easy-to-use, highly expandable, data acquisition and control systems composed of I/O modules and communication interfaces. FieldPoint modules are widely used for instrument measurements, industrial control, and data logging applications. Each module can control up to eight platinum sensors and the system communicates

1 http://www.omega.com/pptst/CY7.html
2 http://www.ni.com/
3 http://www.omega.com/pptst/CY7.html

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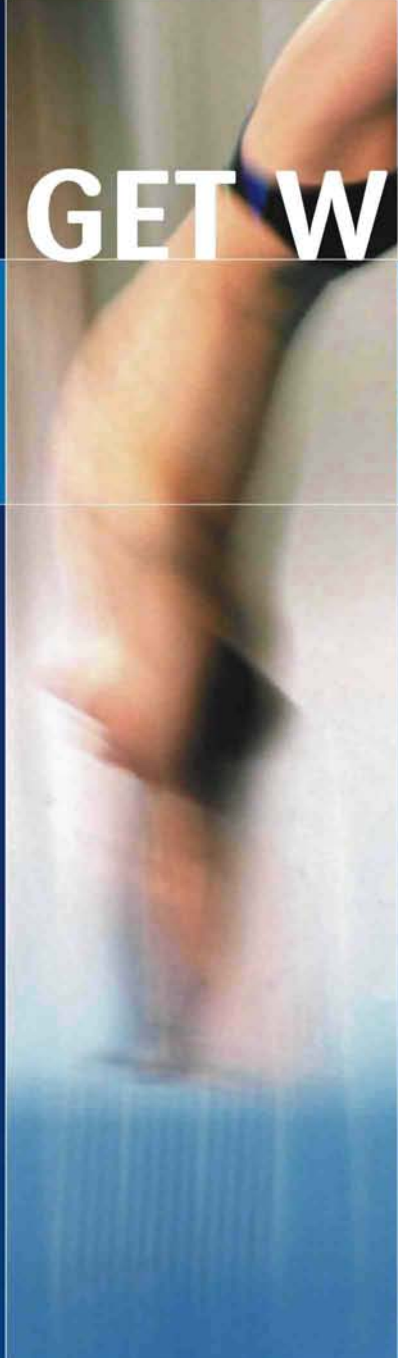
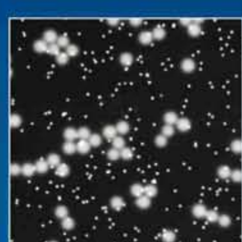
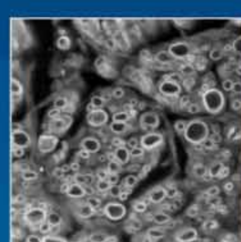


NIH-3T3 cell



Nanoparticles



Rat sciatic nerve

Trypanosoma brucei

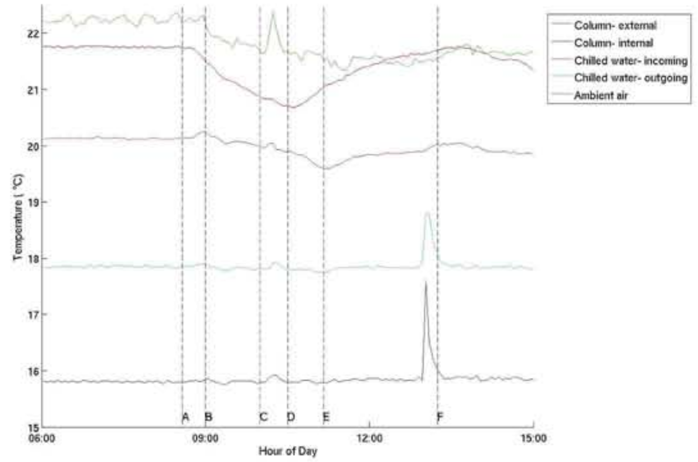


Figure 3: Temperature profiles in Room #2 annotated to reflect various events which occurred during the course of the data acquisition. A) Operator entered room and filled cold trap dewar with LN_2. B) Cold stage loaded into microscope. C) Objective lens on during high magnification calibration setup. D) Objective lens off during low magnification data collection. E) Objective lens left on while operator has left the room. The spike in the chilled water is due to an unscheduled topping off of the chiller tanks with warm water. F) Continuation of date collection with objective lens on.

via a standard Ethernet interface.

We have implemented the system in two microscope rooms, referred to below as room #1 and #2. The dimensions of the rooms are 13'x14' with a ceiling height of 10'. Each room has an adjoining equipment room (4'x14') used to house supporting electronics and computers. The facility is described in detail (O'Keefe *et al.* 2004). Instrumentation common to both rooms is a Tecnai F20 microscope and a 4Kx4K CCD camera. Room #2 has additional equipment associated with a robotic specimen loading system (Potter *et al*, 2004). A schematic of the layout of the sensors in each room is shown in figure 1. Four sensors have been setup in room #1: sensor 1.1 measures the ambient air temperature; sensor 1.2 measures the temperature of the chilled water before it enters the column; sensor 1.3 measures the temperature of the chilled water after it exits the column; and sensor 1.4 was mounted within an existing screw hole on the TEM column that penetrates to a depth of 2". Room #2 has a similar arrangement but has 4 additional sensors: sensor 2.5 is a second ambient air temperature sensor that uses an RTD from National Instruments rather than the Omega RTDs used everywhere else; sensor 2.6 is mounted on the outer surface of the TEM column housing; sensor 2.7 measures the temperature inside the liquid nitrogen dewar which cools the cold trap; and sensor 2.8 is used to monitor the temperature of the cryogenic stage.

The FieldPoint modules are mounted on a 35mm steel DIN rail (McMaster-Carr[4]). In room #1, the modules include a 24 VDC, 120 W DIN-rail mountable power supply, (Omron S82K-O1524), a network module (FP-1601), an RTD module (FP-RTD-122), and a sensor array, all mounted on FieldPoint terminal bases (FP-TB-1). In room #2, there is an additional module, FP-AI-100, that measures the voltage from the cryo-sensors 2.6 and 2.7. The module is supplied by a small current source of 10 µA.

We initially used the LabVIEW software (National Instruments) to prototype and test the system. Subsequently, LabVIEW Professional Development software was used to create a standalone binary application, called EMTEM, that can be installed and run on any computer on a network and does not require LabVIEW. This software is freely available from our website: http:/nramm.scripps.edu/software. In our implementation, the EMTEM software runs on a Windows PC. A configuration file (config.ini) is used to provide the basic settings for EMTEM. These include room and instrument identification information, the data acquisition rate, whether output is written directly to a database or to a simple text file, the database settings if required, email addresses for sending automated alerts, and data pathnames for backup files, error log files and calibration settings. In our implementation, EMTEM writes output to a MySQL database and we usually acquire data at a rate of one measurement every four minutes.

In our laboratory, we have also developed a web based temperature reporting system that reads the temperature measurements from the database and displays the results in a web browser either in tabular or graphical form as shown in figure 2. The data can also optionally be downloaded as a simple tab separated table for further analysis using other software tools. The set of PHP scripts used to produce the graphs and tables are available as part of the publicly distributed system. User inputs to the web based interface include the room name, the time interval between queries, and the set of sensors to be queried.

Results

The system has been operational in our laboratory over the past year. We currently have over 800,000 temperature monitoring records in our database. The total size of these records is under 40 Mb, a fairly modest size when compared to the size of the digital images recorded from the TEMs (one 4Kx4K CCD image is 32Mb).

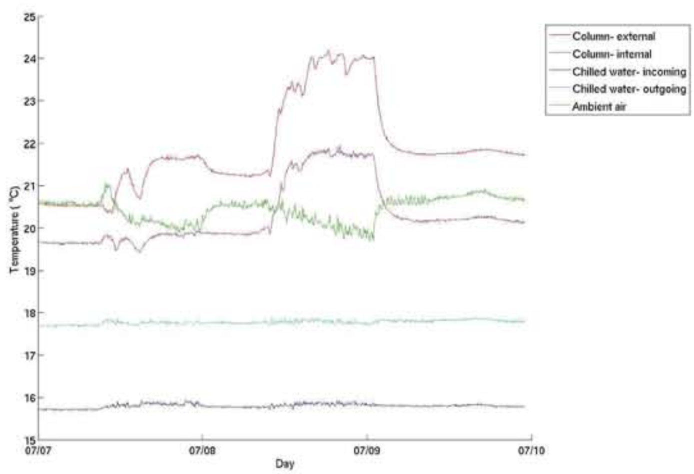
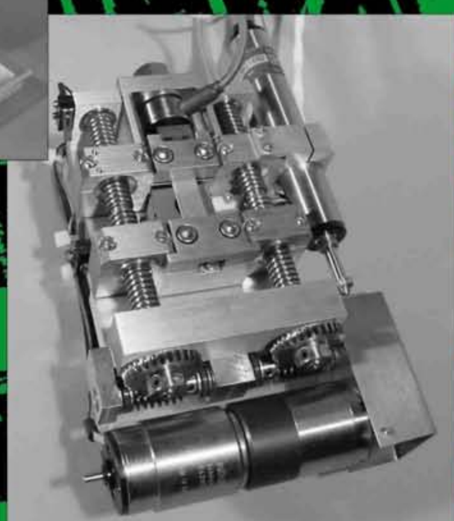


Figure 4: Temperature profiles in Room #2 covering a period of time when the instrument was used in two consecutive experiments, first operating at 120KeV during the day 07/07 and then at 200KeV on 07/08.

The baseline temperature stability of the rooms is shown in figure 2 for room #2. These measurements were made over a 12 hr period when there was no activity in the room. The air temperature is very stable with a temperature drift of less than 0.01 °C/hr and maximum fluctuations less than 0.06 °C/min and the ambient air temperature is within 1 °C of the column temperature. The temperature of the cooling water supply to the microscope is also very stable with fluctuations of less than 0.02 °C/hr. Except for the ambient air temperature fluctuations, these temperature variations are well within the specifications stipulated by the manufacturer. Our experience suggests that the air temperature fluctuation specification would be difficult to achieve in practice, particularly if personnel are present in the microscope room.

4 http://www.mcmaster.com/



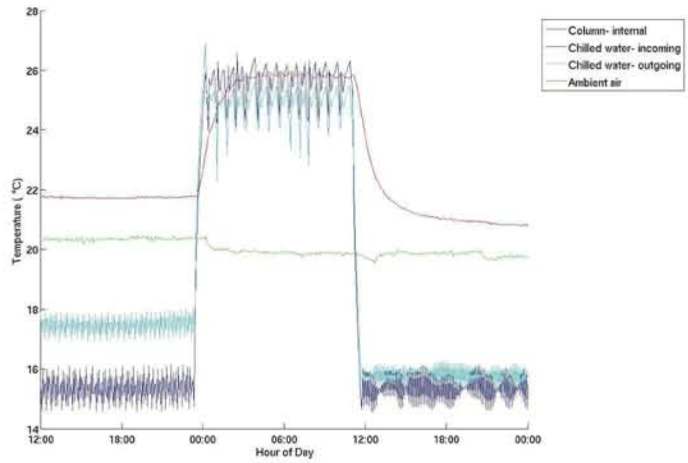


Figure 5: Temperature profile during periods when the equipment malfunctioned due to a failure of the air conditioning in the room housing the chiller equipment.

The temperature plot displayed in Figure 3 shows a fairly typical variation in temperature when the instrument is in use. The plot shown covers an 11 hour period during a typical day of microscope operation in room #2. The plot has been annotated to reflect the various events that occurred during the course of the data acquisition. These include entry into and exit from the room by the operator, addition of liquid nitrogen to the cold trap, insertion of the liquid nitrogen cooled cold stage, changes in magnification on the microscope that lead to the objective lens being turned on or off, and an unexpected and unscheduled maintenance call to the equipment room. These results show that filling the cold trap and loading the cold stage into the instrument results in a ~1°C drop in the temperature on the outside of the column over a period of about 2 hours. However, the corresponding variation of the temperature at the sensor inside the column is less than 0.5°C. As expected, turning on the objective lens of the instrument (magnifications above 2100x) puts a heat load on the column that compensates for the cooling caused by the cryogens and leads to a gradual 1°C increase in the temperature over several hours. The unusual spike in the chilled water inlet temperature occurred as the result of an unscheduled maintenance call to the equipment room housing the chillers, during which the chillers were topped up with water that was warmer than the chilled water. The recovery time of the chiller is less than 30 minutes and the column temperature remains relatively stable throughout this event.

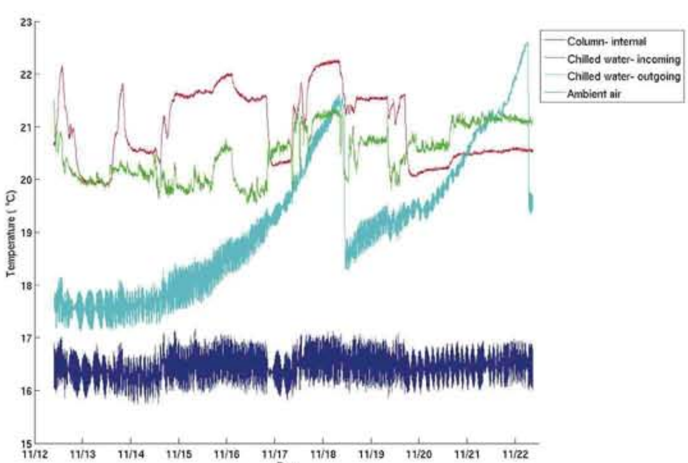


Figure 6: Temperature profile when there was a blockage in the cooling water lines for the CCD camera.

The heat load on the column is very dependent on the operating voltage of the instrument as illustrated in Figure 4, which covers a three day period during which the instrument was operated at 120KeV for approximately 14 hours, unused for 10 hours and then operated at 200KeV for 14 hours. The higher accelerating voltage places a considerably higher load on the column, resulting in temperatures at the sensor inside the column approximately 2 degrees higher than at 120KeV. The rise in temperature is again correlated to the load caused by turning on the objective lens. It can be noted however that even at the higher voltage with the objective lens turned on, the temperature of the internal column sensor is never more than 2 degrees above the ambient air temperature, conditions within the manufacturer's environmental specifications.

The temperature monitoring system has also provided a useful means of discovering potential malfunctions in the overall system. Figure 5 displays the temperature profile during a period when the ventilation cooling system failed in the room where the chillers reside. The air cooled chillers were unable to maintain their set temperatures until the problem was resolved. Figure 6 is another example of equipment malfunction. In this case one of the cooling lines to the CCD camera was blocked, limiting the flow rate through the instrument. The temperature of the outgoing water from the instrument increased by 4 °C, and then the line was manually cleared and the temperature dropped immediately. The line quickly became blocked again and the temperature again increased dramatically before the system was cleared again.

Discussion

We have found the temperature monitoring system to be useful in a variety of ways. It provides a record of environmental conditions during data collection so that data compromised by unstable environmental conditions can be eliminated from further processing. It has also provided us with the capability to understand how fluctuations in environmental effects might affect the quality of the data. The temperature monitoring system also provides an effective early warning system for instrument or environment malfunction in that it is capable of sending out email or pager alerts when temperatures stray outside set limits.

The system as described is easily constructed using off the shelf hardware components and control software which we are freely distributing (http://nramm/scripps/edu/software). We estimate that the entire system can be constructed and installed for less than $1500. The system could be readily extended to include other temperature sensors (*e.g.* incoming air temperature) as well as additional sensors such as a voltage monitor for the incoming mains, vibration sensors, infrared sensors to monitor room entries/exits etc. ■

Acknowledgements:

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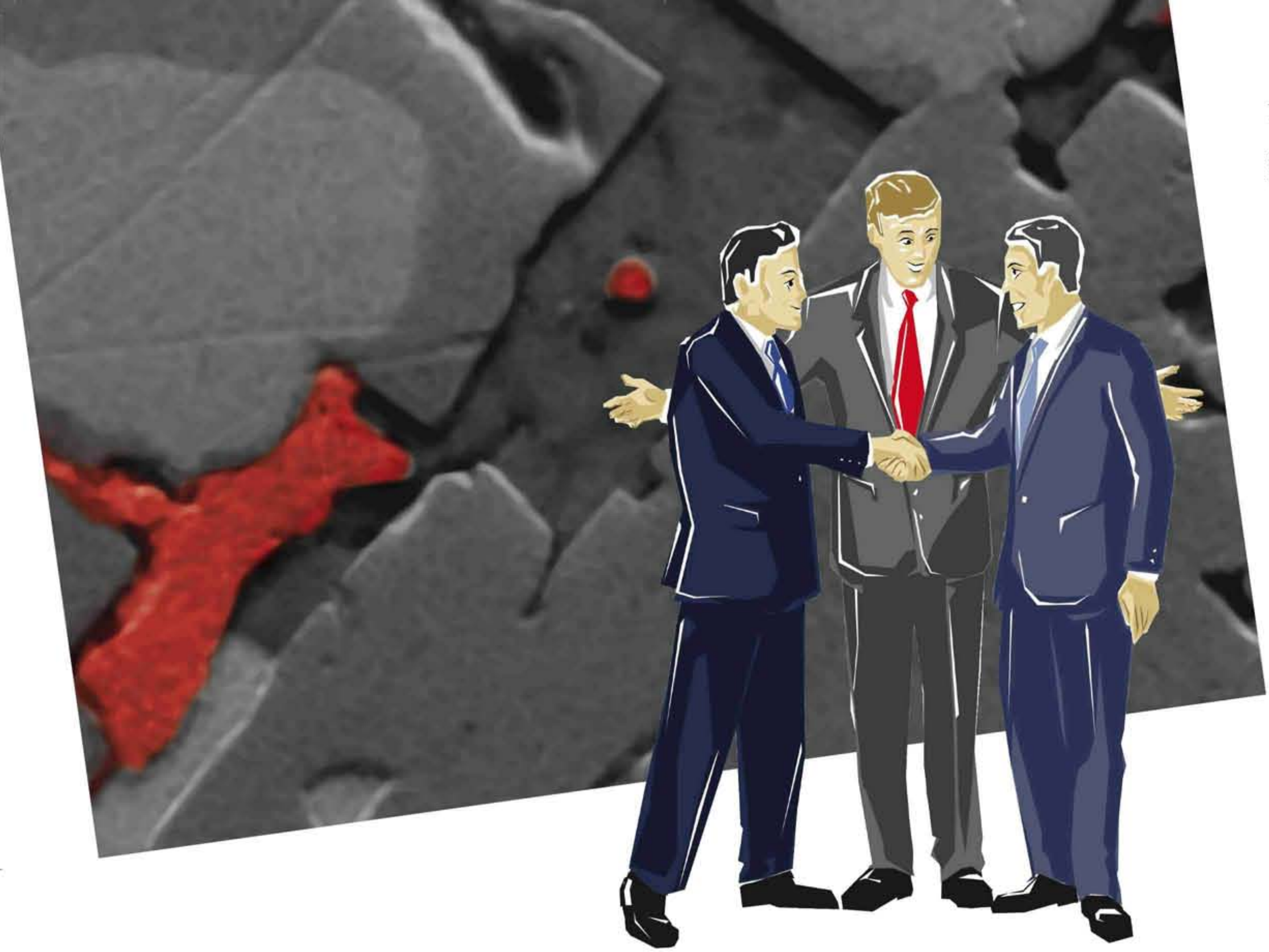

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