

AWARDS



Vittorio de Nora Award

The Honors and Awards Committee is soliciting nominations for the Vittorio de Nora Award in Electrochemical Engineering and Technology. This award will be presented at the Spring 2000 Meeting of the Society in Toronto, at which time the recipient will deliver an award address on a subject related to the contributions for which the award was received.

The recipients shall be distinguished for outstanding contributions to electrochemical engineering and technology. The award consists of a gold medal and a cash prize of \$7,500.

This is one of the Society's most prestigious awards, and all members are urged to give serious consideration to nominating possible candidates. Nomination forms are available from ECS Headquarters or the Subcommittee Chairman: Jennifer Bardwell, National Research Council of Canada, Bldg. M-50, Rm. 190, Ottawa, ON, Canada K1A 0R6; tel: 613.993.8572, fax:

613.990.0202, e-mail: jenniferbardwell@nrc.ca. Eight copies of each nomination must be received by the beginning of the Spring 1999 ECS Meeting.



The Electrochemical Society Fellow

Nominations are now being accepted for the 1999 Class of Fellows of The Electrochemical Society. Selection as a Fellow is one of the highest honors bestowed on a member of the Society, acknowledging that the recipient is distinguished by outstanding contributions to the advancement of science and technology in the areas of electrochemistry and/or solid-state sciences, leadership in the advancement of electrochemical or solid-state science and technology, and current active participation in the Society's affairs. All members are urged to give serious consideration to nominating possible candidates.

Those candidates selected as Fellows will be recognized by the awarding of a certificate and a lapel pin at the Society Awards and Recognition Session at the

1999 Fall Meeting of the Society, an announcement in *Interface*, listing as a Fellow when an author of a **Journal** or **Letters** article, and notation on the identification badge at Society Meetings. Fellows are elected at a rate of up to 20 per year. Deadline for submission of candidates is January 15, 1999.

For more information or for nomination forms, please contact: ECS Headquarters or Dr. Barry R. MacDougall, National Research Council, Inst. for Environmental Chemistry, Ottawa, ON, Canada K1A 0R6; tel: 613.993.8573, fax: 613.941.2529, e-mail: barry.macdougall@nrc.ca.



Henry B. Linford Award

Nominations are now being solicited for the Society's Henry B. Linford Award for Distinguished Teaching. The ninth presentation of this Award, which consists of a silver medal, a scroll, and a cash prize, will be made at the Spring 2000 Society Meeting in Toronto, Canada.

Fellows of The Electrochemical Society

1990

Allen J. Bard
Robert B. Comizzoli
Glenn W. Cullen
Theodore I. Kamins
Paul C. Milner
Edward H. Nicollian
Robert A. Osteryoung
Arnold Reisman
Lubomyr T. Romankiw
Geraldine C. Schwartz
Ben G. Streetman
J. Bruce Wagner, Jr.

1991

Theodore R. Beck
Elton J. Cairns
Bruce E. Deal
Werner Kern
William A. Pliskin
Charles W. Tobias
Rolf Weil

1992

Richard C. Alkire
Vittorio de Nora
Jerome Kruger
Barry Miller

Dennis R. Turner
Jerry M. Woodall

1993

Richard P. Buck
Larry R. Faulkner
Dennis W. Hess
Vik J. Kapoor
Rolf H. Muller
Carlton M. Osburn
Robert A. Rapp
George L. Schnable
Y. H. Wong
Petr Zuman

1994

George K. Celler
Sung-Nee (George) Chu
John P. Dismukes
Richard B. Fair
Adam Heller
Richard A. Oriani
Boone B. Owens
Wayne L. Worrell

1995

Fred C. Anson
Laurence D. Burke

Brian E. Conway
Robert P. Frankenthal
Karl M. Kadish
Digby D. Macdonald
Gleb Mamantov
Florian Mansfeld
Royce W. Murray
John Newman
Yutaka Okinaka
Howard W. Pickering
George Rozgonyi
Mordechay Schlesinger
Karl E. Spear

1996

John M. Blocher, Jr.
Hans K. Bohni
Der-Tau Chin
Hugh Isaacs
Wolfgang Joachim Lorenz
S. J. Pearton
Subhash C. Singhal
Venkataraman Swaminathan

1997

James A. Amick
Denis Noel Buckley

Michel J. Froment
Koji Hashimoto
Chung-Chiu Liu
Ed McCafferty
Theodore D. Moustakas
Shyam P. Murarka
Stella W. Pang
Joachim Walter Schultze
James D. Sinclair
Norman L. Weinberg
Lawrence Young

1998

Huk Y. Cheh
Donald E. Danly
Dennis H. Evans
Funio Hine
Dennis C. Johnson
Zoltan Nagy
Katsumi Niki
Jun-ichi Nishizawa
Fan Ren
Antonio J. Ricco
David A. Shores
William H. Smyrl
George Thompson

The recipient shall be distinguished for excellence in teaching in subject areas of interest to the Society. The award consists of a silver medal and a cash prize of \$2,500.

Nominations for this Award must be accompanied by supporting documents such as a biography; a list of recent courses taught; a list of publications, including any textbooks; professional activities; a list of former students and their affiliations; and other appropriate supporting materials. Letters of support by distinguished colleagues and former students are also encouraged.

To obtain a nomination form, contact the Subcommittee Chairman or ECS Headquarters. Please send eight copies of the nomination form and all documents to: V. Kapoor, College of Engineering, University of Toledo, Toledo, OH 43606; tel: 419.530.8000, fax: 419.530.8006, e-mail: vkapoor@eng.utoledo.edu. Eight copies of each nomination must be received by the beginning of the Spring 1999 ECS Meeting.



Battery Division Research Award

Members of The Electrochemical Society are invited to nominate candidates for the Research Award of the Battery Division. The Award recognizes outstanding contributions to the science and technology of primary and secondary cells and batteries, and fuel cells. Of particular interest are advances in the understanding of mechanisms, introduction of a new theory, and development of new materials.

The Award consists of a check for \$1,000, lifetime membership in the Battery Division, and a scroll describing the Award.

Nominations, in the form of a letter detailing the accomplishments of the nominee(s), should be submitted to the Chairman of the Research Award Committee before March 15, 1999: K. M. Abraham, Covalent Associates, 10 State Street, Woburn, MA 01801; tel: 617.938.1140, fax: 617.938.1364. The letter should be accompanied by a list of all supporting publications, at least one of which must have appeared in the **Journal** or another Society publication.



Battery Division Technology Award

Members of the Society are invited to nominate candidates for the Technology Award of the Battery Division. The Award honors members of the Society who have made noteworthy practical contributions to battery and/or fuel cell technology. Their contributions may consist of novel/practical designs, the development of new or established systems with improved performance or safety, improved manufacturing methods, or improvements in the environmental aspects of battery/fuel cell technologies.

The Award consists of a check for \$1,000, lifetime membership in the Battery Division, and a scroll describing the Award.

Please send the name(s) of your suggested candidate(s) to the Technology Award Committee Chairman: Dr. Arabinda Dey, 17 Shanley Way, Buzzards Bay, MA 02532; tel: 508.759.4617, fax: 508.759.4617. Your letter should indicate the technological contributions of the candidate(s) accompanied by a list of

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relevant publications, with subject titles. At least one of these papers should have appeared in a publication of the Society. Deadline for application is March 15, 1999.



Electrodeposition Division Research Award

The Research Award of the Electrodeposition Division, presented annually, recognizes recent outstanding achievement or contribution in the field of electrodeposition. The Award, consisting of a scroll and \$2,000, will be given to the author or co-authors who have published a paper or papers in one of the Society's publications at the Fall 2000 Society Meeting in Hawaii.

Please submit nominations in letter detailing the accomplishments of the nominee accompanied by a list of supporting publications with titles to: Madhav Datta, IBM Corporation TJ Watson Research Center, Room 40-025, P.O. Box 218, Yorktown Heights, NY 10598; tel: 914.945.2183, fax: 914.945.2141, e-mail: madhav@watson.ibm.com. The nomination deadline is April 1, 1999.



Manuel M. Baizer Award

Nominations are invited for the 2000 Manuel M. Baizer Award of the Organic and Biological Electrochemistry Division. This Award, sponsored by The Electrosynthesis Company, Inc. and by Monsanto Company, recognizes individuals who have made outstanding contributions to the electrochemistry of organic and organometallic compounds, carbon-based polymers, and biomass, whether fundamental and applied.

The Baizer Award will be presented at the Spring 2000 Society Meeting in Toronto. The recipient will receive an honorary scroll and \$1,000, and must present an award lecture at the Organic and Biological Electrochemistry Division Business Meeting.

Previous recipients of this Award include Professor Tatsuya Shone of Kinki University in Japan (1994); Professor Henning Lund, of Aarhus University in Denmark (1996); Dr. Sigeru Torii, of Kyoto University, and Dr. Hans Schaeffer, of the University of Münster (1998).

Submit your letter of nomination, listing the accomplishments and supporting publications of your nominee to

Professor D. Evans, Department of Chemistry, University of Delaware, Newark, DE 19716; tel: 302.831.6770, fax: 302.831.6335, e-mail: dhevans@udel.edu.

The deadline for submission of nominations is January 15, 1999.

The European Local Section Volta Award

The European Local Section has established the "Alessandro Volta Medal," an award to recognize outstanding contributions in electrochemistry and solid-state chemistry and technology in Europe. The award consists of a silver medal and a check for \$500. The recipient will be required to attend a European Local Section sponsored meeting, at which the award will be presented, and deliver a lecture on a subject related to the contribution for which the award has been granted. Nomination forms may be obtained from ECS Headquarters or the Award Chairman. Nominations should be submitted to: Professor B. Scrosati by March 1999, at: Department of Chemistry, University "La Sapienza," Piazza A. Moro 5, 00185 Rome, Italy; tel: +39-06-4462866, fax: +39-06-491769, e-mail: scrosati@axrma.uniroma1.it. ■

Analysis of the Structure and Magnetic Properties of New Multicomponent Transition Metal Nitride Compounds

A Summary Report to The Electrochemical Society for the 1997 F. M. Becket Memorial Award

by K. Scott Weil

A class of solid-state materials which has begun to receive increased attention in the materials community recently is the nitrides. However, there is a general scarcity of nitride compounds and consequently, a relative lack of knowledge about their chemical and physical properties. These materials are virtually non-existent in nature and must be created under controlled conditions in the laboratory; only several hundred nitrides are even known. In spite of this relatively small number, the nitride family includes materials of technological importance, such as silicon nitride (Si_3N_4) used as an insulating coating on semiconductor circuits¹, gallium nitride (GaN) in semiconducting lasers for optical storage devices², aluminum nitride (AlN) in electronic packaging, and tantalum nitride (TaN) in refractory and diffusion-resistant coatings which can withstand a range of corrosive high-temperature environments.³

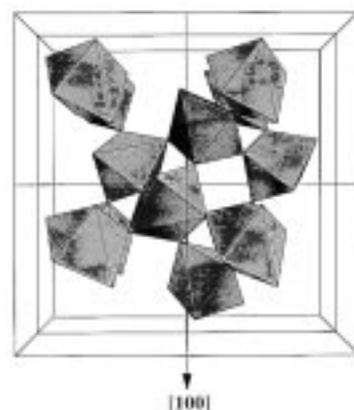
Unfortunately, the chemical properties of nitrogen present a number of difficulties in the synthesis of nitride compounds, in particular multicomponent nitrides. The primary challenge lies in the fact that most of these compounds have small free energies of formation due in part to the strong triple bond of N_2 , one of the potential decomposition products. Thus, the standard solid-state approach of forming compounds from a mixture of their constituents at high reaction temperatures is generally precluded because these nitride phases tend to be stable only at lower temperatures. Our research has focused on the development of lower temperature synthesis strategies to produce multicomponent nitride compounds. We used a chelation technique to prepare mixed-metalloorganic precursors which can be ammonolyzed at moderate temperatures, 500-900°C. This temperature range is much lower than that typically employed in conventional reactions, permitting subsolidus metastable regions of a given phase diagram to be investigated which would otherwise be inaccessible. Due to the general lack of research on multicomponent transition metal nitrides, we focused our efforts on searching for new and potentially exciting combinations of first row and later row transition metals with



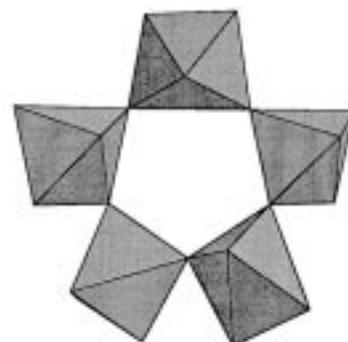
Fig. 1. The proposed unit cell structure of $\text{Ni}_2\text{Mo}_3\text{N}$. The dark gray, large spheres represent the molybdenum atoms; the light gray, medium size spheres represent the nickel atoms; and the small, dark gray spheres represent the nitrogen atoms.

nitrogen. Several new nitride compounds were synthesized and then characterized this summer at the Arrhenius Laboratory in Stockholm University; our most recent findings on the crystal structure and the electrical and magnetic properties of one of these, $\text{Ni}_2\text{Mo}_3\text{N}$, are summarized here.

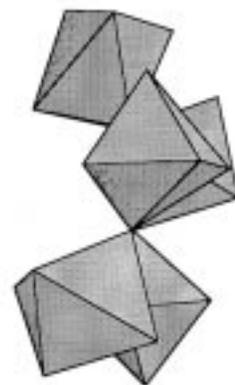
$\text{Ni}_2\text{Mo}_3\text{N}$ was prepared in powder form using the chelated metalloorganic precursor approach, the details of which have been described elsewhere.⁴ Two sets of X-ray diffraction patterns were used in the structural analysis work. One pattern, used for indexing and lattice parameter measurements, was generated by the Guinier-Hägg technique using $\text{Cu K}\alpha_1$ radiation over a 2θ range of 0-90°. An internal silicon standard was incorporated with the sample prior to conducting the diffraction experiment in order to prepare accurate lattice parameter measurements. A series of computer programs, TREOR⁵, VISSER⁶, DICVOL⁷, and PIRUM⁸, were then used to determine the Bravais lattice and indexing sequence and to refine the lattice parameters of the compound. A second set of X-ray diffraction data, which was used for structural refinement, was collected with a STOE STADI/P diffractometer using the following step scan settings: $\text{Cu K}\alpha_1$ radiation, 5-130° 2θ range, 0.02° step size, and 325 second collection time. The Rietveld refinement program, FULLPROF⁹, was applied to refine the crystal structure of the nitride, including the following



2a



2b



2c

Fig. 2. Polyhedral representations of the NMo_6 octahedra in the $\text{Ni}_2\text{Mo}_3\text{N}$ structure. Fig. 2a displays the coordination of each nitrogen atom by six molybdenum atoms, forming NMo_6 octahedra which share corners. The corner-sharing arrangement of these octahedra into pentagonal rings is illustrated in Figs. 2b and 2c. Note in Fig. 2c that the rings are not flat, but convoluted with two opposing octahedra pointing to the right in this side view and the other three pointing to the left.

variables: space group, atomic parameters, and nitrogen stoichiometry. The diffraction peak profile shape was represented by the modified pseudo-Voigt function, with profile asymmetry intro-

duced by employing a multi-term Simpson's rule integration. Atomic thermal displacements were assumed to be isotropic. Crystal structure graphics and geometric calculations were prepared using Ca.R.Ine software.¹⁰ Temperature dependent four-probe resistance measurements of $\text{Ni}_2\text{Mo}_3\text{N}$ were made on a sintered sample measuring $9.51 \times 3.03 \times 0.45$ mm in size. The measurements were conducted over a temperature range of 1.5 to 300°K using an EM P13 DC picovoltage source, a Keithley 220 programmable current source, and a Hewlett-Packard 3458A multimeter. A Lake-Shore 7130 weak-field a.c. susceptometer was used to measure the magnetic susceptibility of $\text{Ni}_2\text{Mo}_3\text{N}$ over a temperature range of 11 to 300°K in an applied field of 250 A/m and at a frequency of 125 Hz. The data were corrected for the diamagnetic contribution of the sample holder.

The crystal structures of nearly all ternary transition metal nitrides can be classified as one of three types: (1) layered hexagonal or rhombohedral structures such as MnWN_2 ¹¹; (2) cubic, η -nitride structures such as $\text{Fe}_3\text{W}_3\text{N}$ ¹²; or (3) cubic, anti-perovskite structures, such as Fe_3NiN and Mn_3CuN .¹³ $\text{Ni}_2\text{Mo}_3\text{N}$ actually crystallizes in a structure-type previously assumed only by transition metal-metalloid nitride combinations, such as V-Ga-N,¹⁴ and in this context represents a new crystallographic finding for ternary transition metal nitride compounds. The crystal structures of the layered nitrides, the η -nitrides, and the new $\text{Ni}_2\text{Mo}_3\text{N}$ compound all display a common trait: each contains a form of metallic clustering; that is, the two transition metal species in each compound arrange into their own separate polyhedral metal clusters that link together in a periodic arrangement.

The structure of $\text{Ni}_2\text{Mo}_2\text{N}$ is not entirely unique, it crystallizes in an ordered variation of the β -Mn structure, isostructural with that of $\text{Al}_2\text{Mo}_2\text{C}$.¹⁵ Analysis of the X-ray diffraction patterns of $\text{Ni}_2\text{Mo}_3\text{N}$ indicate that it is a cubic compound, in the space group $P4_132$ with a lattice parameter of $a = 6.6340(2)$ Å. The unit cell of $\text{Ni}_2\text{Mo}_3\text{N}$ is shown in Fig. 1. Polyhedral representations of the atomic coordination in $\text{Ni}_2\text{Mo}_3\text{N}$ consistent with the Rietveld refinement and diffraction data are displayed in Figs. 2a-c and 3a-d. As shown in Fig. 2a, the nitrogen atoms of this metal rich compound are octahedrally coordinated by molybdenum, forming very slightly distorted NMo_6 octahedra. These octahedra share corners forming a three-dimensional network. When

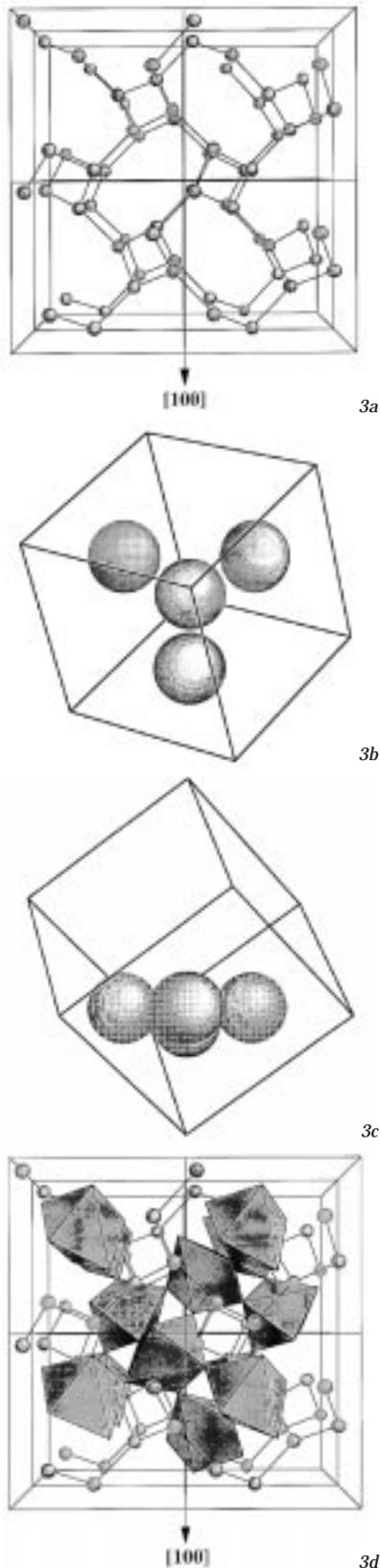


FIG. 3. Representations of the nickel connectivity in the $\text{Ni}_2\text{Mo}_3\text{N}$ structure. FIG. 3a shows the "net-like" linkages between the nickel atoms in this intermetallic nitride. FIGS. 3b and 3c display the triangular arrangement of each nickel atom's three nickel nearest neighbors. Note in FIG. 3c that only the three nearest neighbor atoms are coplanar, with the central nickel atom resting slightly below this plane. The combination of the two interpenetrating lattices in the $\text{Ni}_2\text{Mo}_3\text{N}$ structure is shown in FIG. 3d

viewed along the [322] direction, the corner-sharing NMo_6 octahedra of $\text{Ni}_2\text{Mo}_3\text{N}$, shown in Fig. 2b, appear to form five-membered rings, each containing a small pentagonal-shaped hole. When viewed along the [320] direction, Fig. 2c, the ring is obviously not flat, but assumes an undulated conformation with three octahedra all pointing in one direction and two octahedra pointing in the other. Displayed separately in Fig. 3a, the nickel atoms also form a three-dimensional net-like structure in which each nickel atom is bonded to three adjacent nickel atoms. These three adjacent nickel atoms are coplanar forming an equilateral triangle, Fig. 3b. The central nickel atom, however, does not quite lie in this plane, Fig. 3c, but is centered 0.4448 Å below it. This "fishing-net" lattice of nickel atoms interpenetrates within the interstices of the NMo_6 lattice structure, Fig. 3d.

Four probe dc resistivity measurements carried over a temperature range of 1.5 to 300 K were conducted on a densified bar of $\text{Ni}_2\text{Mo}_3\text{N}$. As seen in Fig. 4, between 49.8 and 300 K the resistivity of $\text{Ni}_2\text{Mo}_3\text{N}$ displays a linear temperature dependence and a positive temperature coefficient of resistance of $3.23 \times 10^{-9} \Omega \cdot \text{m}/\text{K}$, which is characteristic of metallic electrical behavior. In fact, the resistivity in this temperature range is relatively low and is comparable to that of a metallic conductor such as nichrome wire. Below 49.8 K, the resistivity curve changes slope, flattening until 5.9 K at which point a sharp decrease is observed, as seen in the inset in Fig. 4. The change in slope at 49.8 K is likely due to an antiferromagnetic to paramagnetic transition, with $T_N = 49.8$ K. The lower temperature trend, $T < 5.9$ K, is indicative of a possible Meissner effect in the sample, although during testing, the temperature was never decreased to a point low enough that zero resistivity was observed. Interestingly, the related carbide, $\text{Al}_2\text{Mo}_3\text{C}$, which also crystallizes in the β -manganese structure, is a known superconductor with a critical temperature of $T_c = 10.0$ K¹⁵ and a critical field of $H_c = 156$ kOe at 1.2 K.¹⁶

The magnetic measurements were conducted on the as-ammonolyzed powders over a temperature range of 11 to 323 K, which was not low enough to confirm indications of a Meissner phenomenon observed during the resistivity measurements. In all cases, susceptibility data collected at different

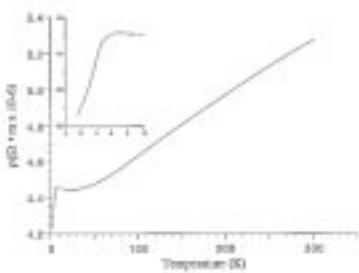


Fig. 4. Resistivity data for $\text{Ni}_2\text{Mo}_3\text{N}$ as a function of temperature. Displayed in the inset is the electrical behavior at low temperatures.

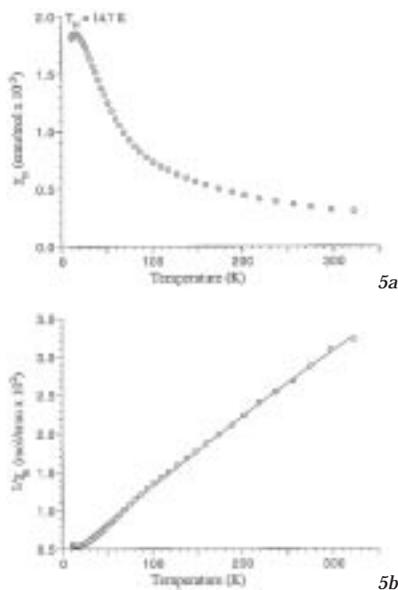


Fig. 5. (a) The molar susceptibility and (b) the inverse molar susceptibility of $\text{Ni}_2\text{Mo}_3\text{N}$ as a function of temperature. The solid line shows the fit of the data to the Néel equation for a two-sublattice antiferromagnet.

frequencies and fields yielded identical curves. Figures 5a and 5b show the molar susceptibility and inverse molar susceptibility curves as a function of temperature for $\text{Ni}_2\text{Mo}_3\text{N}$. The sample displays a fairly well-defined antiferromagnetic ordering peak at 14 K and above that temperature the behavior appears to be paramagnetic. The fact that this T_N differs from that observed in the conductivity measurements may be due to an inadvertent loss of nitrogen from the compound that could have occurred during the thermal consolidation of the conductivity sample. This will be examined in future magnetic studies on $\text{Ni}_2\text{Mo}_3\text{N}$. At present, we are conducting neutron diffraction experiments at the Studsvik Neutron Research Laboratory in Sweden at both room temperature and 10 K to examine magnetic ordering phenomenon in $\text{Ni}_2\text{Mo}_3\text{N}$.

We are also preparing additional conductivity specimens to test at low temperatures ($T < 1.5$ K) to determine if the electrical behavior is repeatable and if the compound does display supercon-

ductivity. What is most intriguing about the possibility of superconductivity in this particular compound is that it would co-exist with magnetic ordering, a combination of properties which is not often seen in superconducting materials.¹⁷ Since the superconducting critical temperature, $T_c < 1.5$ K, is much lower than the ordering temperature, $T_N \sim 14$ K, this material may be ideal for investigating the interplay between magnetism and superconductivity. ■

Acknowledgments

The author would like to thank ECS for the F. M. Becket Memorial Award which provided the opportunity to work abroad. The author would like to acknowledge the assistance of Björn Lundqvist (Royal Institute of Technology) and Lars Göethe (Stockholm University) in conducting the resistivity measurements and preparing the Guinier-Hägg data, respectively. He is extremely grateful to his hosts Dr. Jekabs Grins (SU) and Dr. Gunnar Svensson (SU) for their endless support and advice on this project and to the staff at the Department of Inorganic, Physical, and Structural Chemistry at Stockholm University for their hospitality. Much appreciation also goes to his advisor, Dr. Prashant Kumta, of Carnegie Mellon University, for his guidance on the author's thesis work.

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About the Author

K. Scott Weil is currently developing and studying a metalloorganic precursor approach to the synthesis of new nitride materials for his PhD under the direction of Dr. Prashant N. Kumta at Carnegie Mellon University. He is also a consultant to the Westinghouse Electric Corporation on a new ceramics processing technique that he developed while there as an engineer.