ON THE ORIGIN OF "SYNTHETIC METALS": HERBERT MCCOY, ALFRED UBBELOHDE, AND THE DEVELOPMENT OF METALS FROM NONMETALLIC ELEMENTS

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Introduction

Unlike typical saturated organic polymers, conjugated polymers (Figure 1) are a class of organic semiconducting materials that exhibit enhanced electronic conductivity in their oxidized or reduced states (1, 2). As such, these materials combine the electronic properties of classical inorganic materials with many of the desirable properties of organic plastics, including mechanical flexibility and low production costs. This combination of properties has led to considerable fundamental and technological interest, resulting in the current field of organic electronics and the development of a variety of modern technological applications. Common applications include sensors, electrochromic devices, organic photovoltaics (OPVs), organic light-emitting diodes (OLEDs), and field effect transistors (FETs) (1-7). In addition, the flexible, plastic nature of the organic materials used as the active layers in such electronic devices has led to the realistic promise of flexible electronics in the near future (4-7).

Typical discussions of the history of these materials generally begin in the mid-to-late 1970s with the collaborative work of Hideki Shirakawa, Alan G. MacDiarmid, and Alan J. Heeger on conducting polyacetylene (8-11). In 2000, the Nobel Prize in Chemistry was awarded to these investigators for their early contributions to the field of conjugated organic polymers and the language of the award further reinforces the common view of the historical origins of these materials, which states that the award is "for the discovery and development of electrically conductive polymers" (12). It is only somewhat recently that reports have begun to present a more complete account of the early history of these materials, with particular attention given to highlighting efforts that predate the commonly cited polyactylene work of the 1970s (13-19). While this previous work may not have been as dramatic or as fully realized as the later polyacetylene studies, many aspects and relationships attributed to the work recognized by the Nobel can be seen in these earlier contributions.

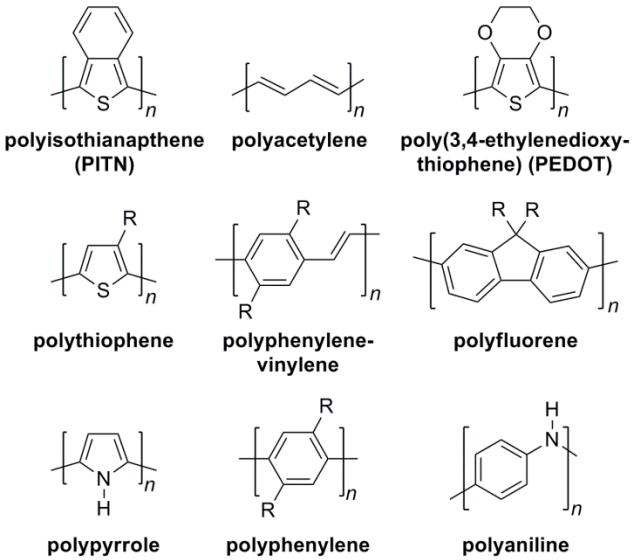

Figure 1. *Common parent conjugated polymers.*

The synthesis and study of conjugated organic polymers dates back to the early 19th century, but it was not until 1963 that the first organic polymer with significant conductivity (10^{-4}-10^{-1} S cm^{-1}) was reported by Donald Weiss and coworkers in Australia on conducting polypyrrole (20-22). This was then followed by the work of Rene Buvet and Marcel Jozefowicz in 1966 on conductive polyaniline powders (up to 30 S cm^{-1}) (14, 18). Two separate reports followed in 1968, which included the first report of doped conductive polyacetylene powders (10^{-4}–10^{-2} S cm^{-1}) by D. J. Berets and Dorian S. Smith, as well as the production of conductive polypyrrole films (7.54 S cm^{-1}) via electropolymerization by Vittorio Bocchi and coworkers (16-18). Shirakawa, MacDiarmid, and Heeger then reported their initial paper on halogen-doped polyacetylene (up to 38 S cm^{-1}) in the spring of 1977 (8). While the conductivity reported in this study was not considerably higher than some of the earlier reports, these authors reported more optimized results later that same year with conductivities up to 560 S cm^{-1} (9, 10). It is this second report that is more significant, as this was the first example of metallic conductivity in an organic polymer. In the following years, additional reports of doped conjugated polymers exhibiting metallic conductivity continued to appear in the literature.

As doped conjugated polymers can exhibit electrical conductivities in the metallic range (>10^2 S cm^{-1}) (23), such doped materials have been frequently referred to as *synthetic metals* (24-27). A primary example of this is Alan MacDiarmid's Nobel lecture entitled "'Synthetic Metals': A Novel Role for Organic Polymers" (26, 27). Common uses of the term can also be found as the title of the Elsevier journal dedicated to this class of materials (28) and in the name of the longstanding International Conference on the Science and Technology of Synthetic Metals (29). The descriptor "synthetic metals" has been in use long enough now that it has become somewhat commonplace and few question how or where this term originated. It was therefore thought worthwhile to review the history of this term as part of ongoing efforts to document a more complete history of conjugated organic materials. An initial communication presenting the origin of the term "synthetic metals" was recently published (19) and the current discussion will expand on this previous report with a more detailed history of the origin and evolution of the term.

In reviewing previous discussions of the origin of this term, it is found that credit is commonly given to Alfred R. Ubbelohde (1907-1988), who began using the term in reference to intercalated graphites as early as 1969 (30, 31). A clear example of this can be found in the biographical memoir of Ubbelohde written by F. J. Weinberg (32) which states:

> Ubbelohde coined the evocative expression "Synthetic Metals" to cover the creation of materials with metallic conduction but formed entirely of such non-metallic atoms as carbon, nitrogen, hydrogen, the halogens and oxygen. This expression was later adopted as the title of an international journal publishing papers on these materials from laboratories all over the world.

In reality, however, the term predates Ubbelohde's work and can be found as early as 1911 in the work of Herbert N. McCoy (1870-1945) (33). As a result, it is with McCoy that we will begin the current discussion before returning to the life and work of Ubbelohde.

Herbert N. McCoy

Herbert Newby McCoy (Figure 2) was born June 29, 1870, in Richmond, Indiana, to Sarah and James McCoy (34, 35). Losing his father at a young age, 13-year-old Herbert worked 10 hours a day to help support the family and held a wide variety of jobs by the time he finished his primary education. After graduating from Richmond High School in 1889, McCoy entered Purdue University to study chemistry under John Ulric Nef (1862-1915). Nef had just left Purdue, however, and McCoy thus began studying under Winthrop E. Stone (1862-1921) (34, 36). By taking special examinations, he was admitted as a sophomore, but had to take time off after a year due to lack of funds. Upon his return, additional examinations gave him credit for work done during his time away to raise money, and he was able to complete his bachelor's degree with only two years in residence (34, 36), finishing in the spring of 1892 (34-36). The following year he continued graduate studies under Stone, during which he was supported by a part-time teaching assistantship. He finished his M.S. in Chemistry in 1893 (34-36), with a thesis on the electrolytic oxidation of glycerin (34, 36, 37).

McCoy then worked as a chemist for Swift and Company in Chicago, but preferred academic work and left after a little more than a year to teach the Department of Science and Mathematics curriculum at Fargo College in North Dakota (34, 35, 38). Founded in 1887 and closed in 1922, Fargo College was a private non-sectarian Christian college (Figure 3) and should not be confused with its Fargo neighbor, North Dakota Agricultural College (founded in 1890 and now North Dakota State

University). McCoy stayed in Fargo for two years, during which he prepared for further graduate work (36). He was attracted to the University of Chicago, as John Nef had recently joined the faculty (34), and he started research work there during the summer of 1895. However, as Nef was not at campus that summer, McCoy began working on a problem in organic chemistry under Julius Stieglitz (1867-1937) (34, 35), before returning to Fargo College for the 1895-1896 academic year. The following summer, he was given a fellowship under Stieglitz and left Fargo to enter the University of Chicago as a full time student (36). He finished his Ph.D. in 1898 (34, 35), with a dissertation entitled "On the Hydrochlorides of Carbophenylimido Derivatives" (39). As jobs were scarce at the time, he remained at the University as a research assistant for Alexander Smith (34).

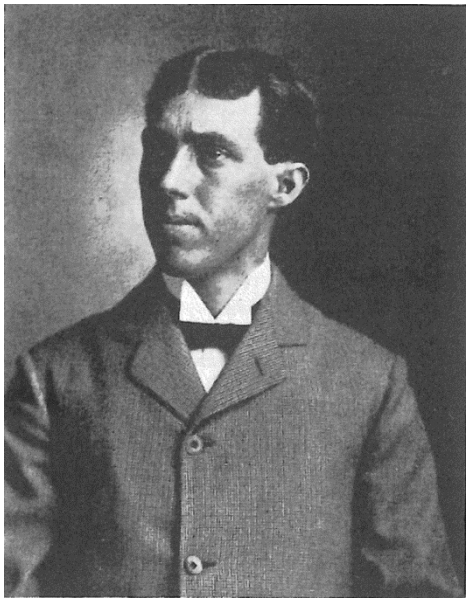

Figure 2. *Young Dr. McCoy (ca. 1900) [From Ref. 34].*

McCoy joined the University of Utah as assistant professor of chemistry in 1899. Because the head of department, Dr. J. T. Kingsbury, had just been appointed University President, McCoy also acted as the unofficial head (34, 36). He then returned to the University of Chicago as an instructor in 1901, replacing Dr. Felix Lengfeld in physical chemistry (34-36).

The University of Utah invited him to return as a full professor in 1903, but McCoy elected to stay at Chicago as an assistant professor (34). He was promoted to associate professor in 1908 and ultimately full professor in 1911 (34, 35). McCoy's research on radioactive chemical elements attracted significant attention from industry, and he

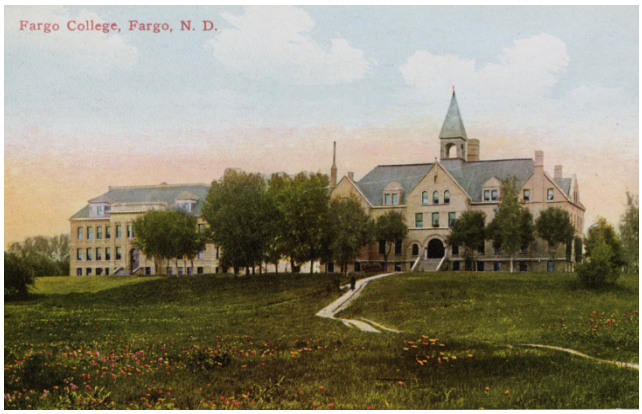

Figure 3. *Fargo College (ca. 1915) [Courtesy of the Institute for Regional Studies Archives, Fargo, ND (rs007656)].*

ultimately left Chicago in 1917 to move to the Carnotite Reduction Company of Colorado, eventually becoming the company's president. He retired from Carnotite when it was sold in 1920, and became vice president of the Lindsay Light Company in Chicago (34-36). McCoy left Chicago in 1927 to move to Los Angeles, although he remained the vice president of Lindsay until his death (34, 35). In Los Angeles, McCoy became a guest in the research laboratory of University of Chicago alumnus Dr. B. A. Stagner, who was now a consulting chemist in Los Angeles. Here, McCoy continued to work on rare-earth problems beginning in 1930 (34). He then built a laboratory over the two-car garage of his Los Angeles home in 1934, where he continued his research on the rare earths, especially with europium and samarium, for the rest of his life (34, 36). His various accomplishments were recognized by receiving the Gibbs Medal in 1937 (36) and being awarded a D.Sc. by Purdue University in 1938 (34). He passed away on May 7, 1945 (34).

Although McCoy was known primarily for his work in rare earth chemistry, he has also been credited with the preparation of what was thought to be the first organic metal (34, 35). This work was reported in three papers over the span of 1911-1912, while he was still on the University of Chicago's faculty, and focused on efforts to produce a metallic species from the electrolysis of tetramethylammonium salts (33, 40, 41). Prior to these publications, the work was initially presented at American Chemical Society Meetings, first at Detroit in June 1909 (40) with a brief write-up in *Science* (42), and then at Minneapolis in December 1910 (33). These efforts were all based on earlier reports of ammonium amalgam, first prepared by Thomas J. Seebeck (1770-1831) in 1808 (43). As the ammonium cation is in many

respects similar to alkali metal cations, it was thought that it could potentially be reduced to generate a neutral ammonium radical, which could theoretically give metallic properties similar to sodium or potassium metal. McCoy explained this hypothesis as follows (33):

> When we come next to consider the behavior upon electrolysis of a salt of a compound basic radical, it is difficult to see wherein its behavior should differ from that of a salt of a metallic element. In this case, as in the other, positive ions are attracted to the cathode, and upon striking the latter can gain electrons. If then the electron theory of the metallic state is as fundamental as it seems to be, the aggregate of such free "neutralized" radicals should be a body having metallic properties; in other words, a "synthetic metal."

In fact, when Seebeck first reduced ammonium carbonate with a mercury electrode (equation 1), a spongy mass was generated, which he believed to be an amalgam of ammonium and mercury (43). This amalgam was then shown to be similar to the mercury amalgams of sodium and potassium, first by Seebeck and later by Humphry Davy (1778-1829) (44). However, the true nature of this amalgam and whether or not it was metallic was heavily debated.

$$NH_4^+ \xrightarrow[Hg]{e^-} Hg/H_4N\bullet \qquad [1]$$

Roughly 100 years later, McCoy and William C. Moore extended these earlier efforts by investigating the electrolysis of organic quaternary amines, rather than just simple ammonium salts (33, 40, 41). Utilizing an electrolytic chamber comprised of silver-plated platinum gauze for the anode and a mercury electrode as the cathode, electrolysis of the salt resulted in the production of a crystalline solid of metallic luster. This solid product closely resembled sodium amalgam and was believed to be a mercury amalgam of ammonium radicals with the general formula $HgN(CH_3)_4$ (40). Although this organic amalgam was not very stable and reacted violently with water, it exhibited electrical conductivities comparable to that of a metal (ca. 7-9 × 10^3 S cm^{-1}) (33, 40, 41). Ultimately, McCoy concluded (33):

> The facts just reviewed, though few in number, seem to me to lend support to this hypothesis, and to lead to the conclusion that it is possible to prepare composite metallic substances, which may be termed synthetic metals, from constituent elements, some of which at least are nonmetallic.

Such ammonium and quaternary ammonium amalgams continued to be of interest up through the early 1970s (45). In 1986, however, the eminent electrochemist Allen J. Bard reported convincing evidence that such ammonium amalgams are more likely Zintl ion salts resulting from the reduction of mercury, rather than reduction of the ammonium cation (46). Thus, the more correct representation of this process would be that given in equation 2 below.

$$NH_4^+ + 4\,Hg \xrightarrow{e^-} NH_4^+(Hg_4^-) \qquad [2]$$

As such, the products generated by these electrolytic processes are not really organic metals, although they were believed to be at the time and do seem to be the origin of the term "synthetic metals." The term was then not used in the literature again until 1969, when Alfred René Ubbelohde of Imperial College described a new class of materials based on intercalated graphite as synthetic metals (28-31).

Alfred R. Ubbelohde

Alfred René Jean Paul Ubbelohde (Figure 4) was born December 14, 1907, in Antwerp, Belgium, the third son of the merchant Francis C. J. Ubbelohde (32). His family moved from Belgium to London when World War I broke out, initially residing in Richmond, Surrey. His education began at Richmond County School in 1918, followed by Colet Court (St. Paul's preparatory school) in 1920, and St. Paul's School. His interests fell equally to chemistry, mathematics, and literature, but he ultimately opted for chemistry and won a scholarship to Christ Church, Oxford, where he graduated with First Class Honors in 1930. He also supplemented his Oxford studies with a B.Sc. Special Chemistry from the University of London, which he passed with First Class Honors as an external student in 1928. Ubbelohde ultimately became a naturalized British citizen in his mid twenties (32).

Following his graduation, Ubbelohde remained at Oxford to spend another five years on postgraduate research, working mostly on hydrocarbon oxidation chemistry with Sir Alfred Egerton (1886-1959) (32, 47). During 1931-1932, he also spent a year at the Institut für Physikalische Chemie, Göttingen, with Arnold Eucken (1884-1950). Following additional research positions at the Royal Institution (1936-1940) and the Ministry of Supply (1940-1945), he became Professor of Chemistry and Head of the Department of Chemistry at Queen's University, Belfast, in 1945. Oxford University awarded him a D.Sc. in 1941 and he was elected a Fellow of the Royal Society in 1951 (32).

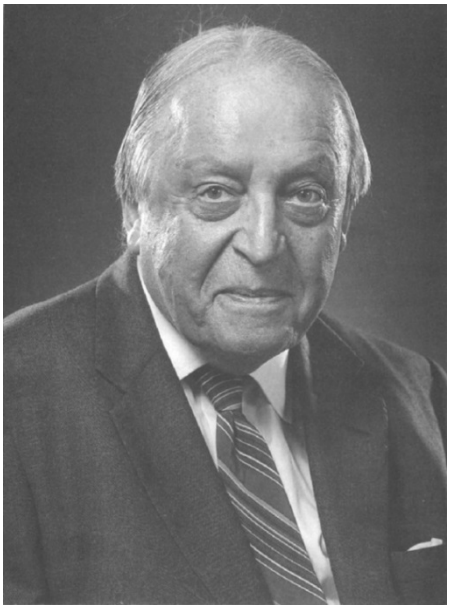

Figure 4. *Alfred R. J. P. Ubbelohde [From Ref. 32; Courtesy of JSTOR].*

In 1954, Ubbelohde moved to Imperial College to become Professor of Thermodynamics in the Department of Chemical Engineering and Chemical Technology. He then became Head of Department in the early 1960s, a post he held until his retirement in 1975. He remained as a Senior Research Fellow until his death on January 7, 1988 (32, 47). With an overall focus of thermodynamics, he spent his career working on a range of subjects including combustion, collision properties of hydrocarbon vapors, carbon, graphite and intercalation compounds, hydrogen in metals, phase transitions, and ionic melts (32). However, it is his work with graphite and its corresponding intercalation com-pounds (Figure 5) that is most critical to the current discussion (28, 29, 48-56).

The various graphite intercalation compounds reported by Ubbelohde during his career exhibited conductivities as high as 2.5×10^5 S cm^{-1} and thus provided the first practical and stable example of a carbon-based species that conducted in the metallic range (28, 29, 48-56). His first paper on these species, which reported graphite intercalated with either bromide or potassium to give metallic conductivities (up to 1.5×10^3 S cm^{-1}), was published in 1951 (48). While these materials would fit the definition of synthetic metals as discussed above, the first time that Ubbelohde uses the term is not until 18 years later when he published a paper on graphite nitrates and bisulphates in 1969, in which the effect of the intercalated ion concentration on conductivity was studied (28). He then followed this with another paper entitled "Electronic Properties of Some Synthetic Metals Derived from Graphite" later that same year (29). In comparison to his previous work, these papers reported significantly higher conductivities (10^4-10^5 S cm^{-1}) and were his first reports of such highly conductive intercalated graphites. It may have been the significantly higher conductivities that led to his use of the term "synthetic metal" to describe the species reported in these papers, or it may be the result of observations that intercalated graphites with high ion concentrations demonstrated greater three-dimensional metallic behavior. For whatever reason, the term then became a mainstay in his writings for the rest of his career (49-56), which thus resulted in the belief that the term was originated by Ubbelohde.

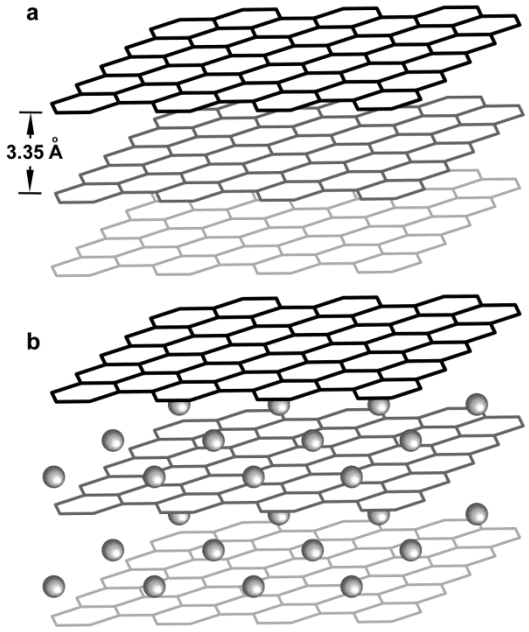

Figure 5. *Schematic representation of native graphite (a) and a simple intercalated compound (b).*

The remaining question at this point is whether Ubbelohde developed the term "synthetic metals" independently or if he learned of the term from McCoy's earlier paper and simply applied it to his own work. This is not possible to answer conclusively, although we can look to the language used by Ubbelohde for clues. It is important to note that Ubbelohde never claims the term as his own, nor does he even introduce or define the term in either of his two 1969 papers. In all cases, he always uses it as if it is a known term that does not require explanation. For example, the very first time we see the term used in his work is in the abstract of his first 1969 paper, where he states (28)

> Various electronic properties confirm a model for the acid salts in which layers of carbon hexagon networks act as macro-cations, separated by layers in which acid anions are linked to additional molecules of acid by hydrogen bonds. In conformity with this model, as the concentration rises, the resistance drops, at first steeply, finally asymptoting to a limiting value characteristic of a synthetic metal.

The term is then used again in the first sentence of the introduction, as follows (28):

> With the development of methods for producing near-ideal graphites by stress annealing pyrolytic graphite, and with improved methods for controlled progressive formation of intercalation compounds from specimens already mounted as electrical conductors, it becomes possible to study variations in charge carrier behaviour in these synthetic metals, in much greater detail than is usually feasible with natural metals.

As can be seen, no definition or explanation of the term is given. At the same time, he does not provide a source for the term and McCoy is never mentioned.

It should be noted, however, that before graphites became such a focus for Ubbelohde, he also studied the production of mercury-ammonium amalgam via the electrolytic reduction of ammonium cations, publishing two papers on the subject in 1951 (57, 58). Thus it seems quite plausible that he would be familiar with McCoy's earlier papers on the topic from 1911. This cannot be confirmed, however, as although Ubbelohde does reference work from the early 1900s in his ammonium amalgam papers, McCoy is again never mentioned and McCoy's paper using the term "synthetic metals" is not referenced. However, it should also be pointed out that even though Ubbelohde summarizes what is currently known on ammonium amalgam in one of his two papers and points to the fact that the topic dates back to the 19th century, none of the seminal ammonium amalgam papers are cited in his work, including no mention of either Seeback or Davy.

Overall, it is just not possible to make a definitive connection between McCoy and Ubbelhde. Still, due to their overlap in research on ammonium amalgams, combined with the fact that Ubbelohde never claims the term "synthetic metals" as his own, it is this author's belief that Ubbelohde learned of the term from the work of McCoy and did not develop it independently. Assuming that this is correct, the reason that Ubbelohde never mentioned, referenced, or acknowledged McCoy will continue to remain a puzzling mystery.

ICSM and New Examples of Synthetic Metals

Through the early 1970s, a variety of additional new materials were discovered that exhibited metallic conductivity, including organic charge-transfer salts, metal chain compounds, and the inorganic polymer poly(sulfurnitride), $(SN)_x$ (Figure 6). As this research spanned a range of scientific disciplines and geography, a specific venue that would bring these interdisciplinary researchers together was desired and thus a workshop was organized in the summer of 1976 in Siofok, Hungary (29). Discussion at this meeting spanned such subjects as charge density wave behavior in the Peierls-Frohlich state and the application of these concepts to charge-transfer salts such as tetrathiafulvalene-tetracyanoquinodimethane (TTF-TCNQ), along with low-dimensional metal-containing materials such as the tetracyanoplatinates, and niobium triselenide ($NbSe_3$). Also considered was the possibility to chemically control the structural and electronic properties of these new materials, including the extent of charge-transfer, the degree and role of band filling, and the effects of extreme anisotropy, interchain interaction, and coulomb repulsion (29).

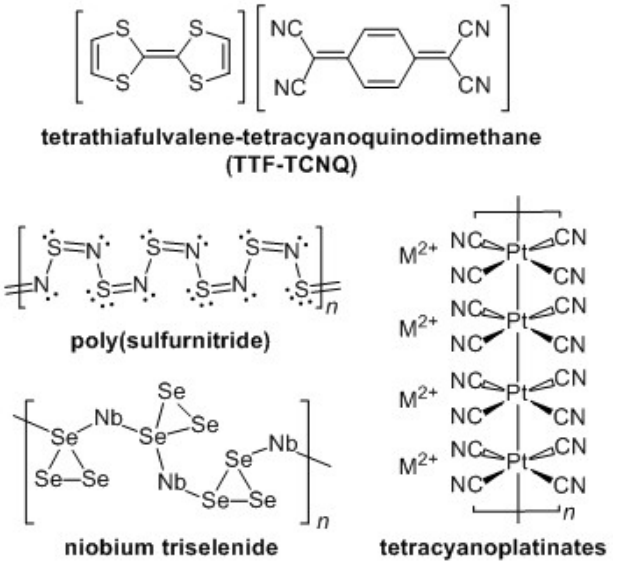

Figure 6. *New examples of synthetic metals.*

This workshop and the discussions that grew out of it resulted in the organization of a long-standing international conference on these materials and issues. This meeting was given the official title of the *International Conference on the Science and Technology of Synthetic Metals*, often referred to by those in the field as just ICSM. The conference was held annually from

1976-1982 and has been held biennially ever since (29). At the time of writing this manuscript, the most recent conference was held in Turku, Finland in 2014, while ICSM 2016 is scheduled to be held in Guangzhou, China.

Following the November 1976 (14, 16, 18) discovery by MacDiarmid, Heeger, and Shirakawa that free-standing films of polyacetylene could be oxidatively doped with bromine and iodine to give high conductivity materials, these results were first reported at the second ICSM conference in New York City (ICSM '77) (29). The production and study of these highly conducting doped polyacetylenes then appeared in the literature starting in the second half of 1977 (8-11), which resulted in the scope of carbon-based materials described by the term "synthetic metals" being expanded to include both intercalated graphites and doped polyacetylenes (59). As such, this broadened the field of synthetic metals and subsequent ICSM conferences included a broader range of polymer and materials scientists. Although Heeger, MacDiarmid, and Shirakawa never used the term in any of their initial polyacetylene papers of 1977-1978 (8-11), MacDiarmid published a review of synthetic metals in 1979 (60). The introduction of this review began with the statement:

> This report is directed toward the very new area of materials science which is concerned with the preparation and characterization of synthetic metals, many of which contain no atoms of any metallic element in their chemical constitution. The three main presently known classes and their potential technological significance will be described.

MacDiarmid then went on to state that these three classes were metallic compounds derived from poly(sulfurnitride), polyacetylene, and graphite (60). MacDiarmid had also previously used the term to describe poly(sulfurnitride) in a radio address in 1977 (61). By 1980, the term "synthetic metals" was starting to be used more and more often to describe doped polyacetylene, and as the field of conducting polymers continued to grow, the term was further expanded to include other doped conjugated polymers. By 1991, MacDiarmid and Arthur Epstein included doped polyparaphenylene, poly(phenylene vinylene), polypyrrole, polythiophene, and polyaniline (Figure 1) in a review of conducting polymers as synthetic metals (24).

A New Dedicated Journal

By October 1979, a new journal was launched by Elsevier dedicated to this growing class of materials, which was aptly titled *Synthetic Metals* (28). In the introduction (62) of the first issue (Figure 7), Editor F. Lincoln Vogel of the University of Pennsylvania (63, 64) described this publication as

> ... a new international journal for the publication of research and engineering papers on graphite intercalation compounds, transition metal compounds, and quasi one-dimensional conducting polymers.

The initial Associated Editors (63) included future Nobel laureate Alan J. Heeger and Wayne L. Worrell (1937-2012), best known for his work in solid electrolytes. Heeger would also go on to take over duties from Founding Editor Vogel to become Editor-in-Chief for the journal in 1984 (65). The initial Editorial Board also included Heeger's collaborator and future Nobel laureate Hideki Shirakawa, as well as Alfred Ubbelohde. The initial issue of the journal also contained Ubbelohde's final published paper on intercalated graphite (56). To date, this is still the only journal dedicated to organic conducting materials.

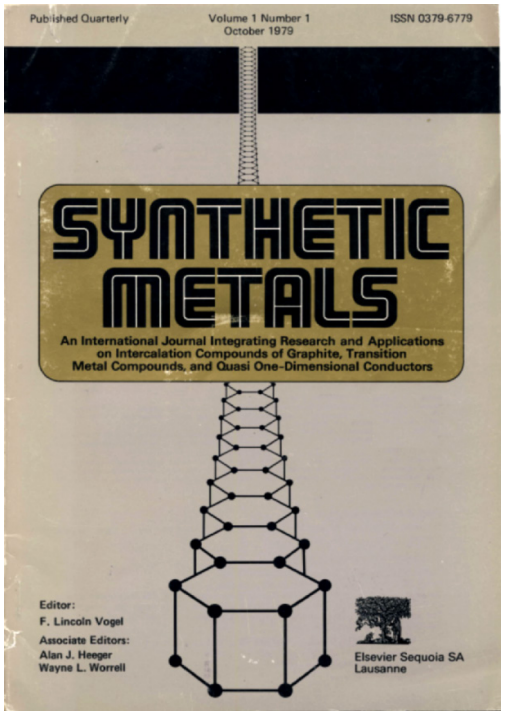

Figure 7. *The cover of the first issue of Synthetic Metals, published October 1979 [Synthetic Metals, Vol 1, Issue 1, Copyright Elsevier and used with permission].*

Conclusions

As hopefully illustrated by the above discussion, the history of synthetic metals can be traced much further

back than commonly viewed by current researchers in the field. In addition, just as our concept of conducting materials has changed drastically over the last 50+ years, the specific materials represented by the term synthetic metals has also changed since its first use in the literature. However, in all cases, these materials have continued to fit the original use by McCoy in 1911 to represent "composite metallic substances ... from constituent elements, some of which at least are nonmetallic" (33).

Acknowledgements

I would like to thank the Department of Chemistry and Biochemistry of North Dakota State University (NDSU) for continued support of my historical research, as well as the NDSU Interlibrary Loan Department, who went out of their way to track down many elusive and somewhat obscure sources. Additional thanks are given to the staff of the NDSU Archives and Institute for Regional Studies for allowing me access to the records of Fargo College and to Vijala Kiruvanayagam at Elsevier for helping me obtain the original cover for the first issue of Synthetic Metals.

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11th International Conference on the History of Chemistry

In summer 2017, the fortieth anniversary of the creation of the Working Party (WP) on History of Chemistry of the European Association for Chemical and Molecular Sciences (EuCheMS) will be celebrated. The general aim of the conferences organized by the WP is to facilitate communication between historically interested chemists and historians of chemistry, and to gather the community on a regular basis. The most recent conferences organized by the WP were held in Rostock 2011 (Pathways of Knowledge), Uppsala 2013 (Chemistry in Material Culture), and Aveiro 2015 (Chemical Biography in the 21st Century).

The 11th International Conference on the History of Chemistry (11th ICHC) will take place from 29 August to 2 September 2017 in Trondheim, a city founded in 997 which served as Norway's capital during the Viking Age. The Norwegian University of Science and Technology (NTNU), which has been the country's centre for technology education since 1910, will host the conference. The conference is sponsored by NTNU, the Research Council of Norway, the Norwegian Chemical Society, the Chemical Heritage Foundation and Sintef Materials and Chemistry.

Keynote lectures will be given by Hasok Chang (University of Cambridge), Maria Rentetzi (National Technical University of Athens) and Anders Lundgren (Uppsala University).

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