

Interstitial compounds

Applications of these materials derive from their hardness, high melting points and, in some cases, catalytic and magnetic properties and superconductivity; yet their electronic structure is still something of a mystery.

Lawrence H. Bennett, Archie J. McAlister and Richard E. Watson

The field of interstitial compounds is a mature one, with an enormous literature.¹ Nevertheless, a question as fundamental as the nature of their electronic structure remains a topic for lively (sometimes acrimonious) debate today. Moreover, industry has yet to tap more than a few of the myriad technical possibilities of these materials. The most commonly exploited property is their great hardness: tons of the interstitial compound tungsten carbide are produced annually for use in grinding and cutting tools. But other properties have seen little commercial application as yet. To cite a few: as a class, they are refractory, yet metallic, and they often have abnormally low work functions, making them excellent electron emitters. This combination suggests, for example, utility as magnetohydrodynamic channel electrodes. Some interstitial compounds, such as NbN, are superconducting, with transition temperatures second only to such champions as Nb₃Sn. Tungsten carbide and related compounds are moderately good catalysts, able to operate in chemically hostile environments, as may be encountered, for example, in fuel cells. Many are respectable ferro- and antiferromagnets. As we will try to indicate, this class of compounds poses fascinating possibilities for the technologist and real challenges for the researcher.

Two examples of applications of these materials are implied in figure 1. The forklift truck in the figure, developed by the Army, combines batteries with hydrogen-powered fuel cells. The batteries

are used for startup and as electrical ballast when high current is drawn for load lifting. The advantage of a hydrogen-fueled lift is constant operation efficiency over a longer operating time. Applications for interstitial compounds in the forklift include the possibilities of using carbide catalysts in the fuel cells and the use of metal hydrides to replace the pressure tanks for hydrogen storage.

What are these materials?

The term "interstitial compound" traditionally refers to combinations of the relatively large transition metals (titanium, vanadium, chromium, manganese, iron, cobalt and nickel) with the small metalloids (hydrogen, boron, carbon and nitrogen). The term might be extended to encompass the oxides, whose chemistry is quite different, and the larger metalloids silicon, sulfur and phosphorous—but we will not consider these here. For many of the compounds of concern to us, the metal atoms occur on simple sublattices with the metalloids occupying interstitial sites within the metallic array. Two such simple structures are illustrated in figure 2: the sodium-chloride structure, in which the octahedral interstitial sites in a face-centered-cubic metal array are occupied by metalloids, and the tungsten-carbide structure, in which one of the two trigonal sites in the unit cell of a simple hexagonal array is occupied by the metalloid. Size considerations generally do not favor the occupation of tetragonal sites. In an alternative picture, one regards the metalloid atom and its nearest metal neighbors as forming a structural unit—the coordination polyhedron, illustrated in figure 2 for octahedral sites—which can be stacked to yield the compound structure.

In general, the structures of interstitial compounds will be simple or complex

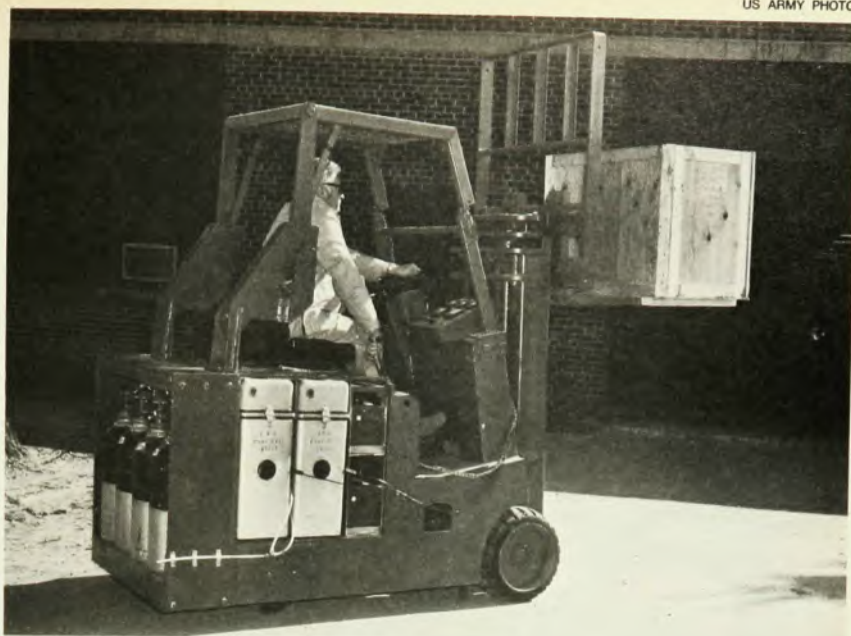
depending on the ratio r of the metalloid atomic radius (r_x) to that of the transition metal (r_{Me}); thus $r \equiv r_x/r_{Me}$. Atomic radii have been used, since the interstitial compounds are normally metallic, and hence at best only partially ionic. If the ratio is less than 0.59, the metal atoms form the aforementioned simple structures—face and body centered cubic, close packed or simple hexagonal. In this case the compounds, called "Hägg" compounds after the discoverer of the radius-ratio rule, are conveniently described in terms of the interstitial-site picture. When the ratio is greater than 0.59, the transition metals and metalloids form complicated structures; an example is the structure of Cr₃C₂, shown in figure 2, which is most easily visualized with the coordination-polyhedron picture.

We should point out that these "interstitial compounds" are, strictly speaking, neither interstitial nor compounds. They are not interstitial in the usual sense, because the metal-atom arrays are almost never those of the pure metal—although the hydrides provide some important exceptions. The materials are really alloys, rather than compounds, in that they often occur over ranges of composition. Consider vanadium carbide as an example. A section of the V-C phase diagram,² somewhat simplified, is shown in figure 3. Note the extensive composition range of the VC phase, from 37 to 47 atomic percent carbon. Vanadium carbide of ideal stoichiometry does not exist at all! Such extensive composition ranges are primarily associated with vacancies on the metalloid sublattice, as in this case, rather than with an atom residing substitutionally on the wrong sublattice. (Ordering of the carbon vacancies in the VC phase can be viewed as introducing new phases in this region, for example V₇C₆.)

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Two applications of interstitial compounds are inherent in the design of this forklift truck, developed by the US Army at Fort Belvoir, Va.

Carbide catalysts are appropriate for its hydrogen-powered fuel cells, and metal hydrides for hydrogen storage may eventually supplant the pressure tanks shown here, to yield a factor of three in volume reduction and some weight savings as well. Figure 1

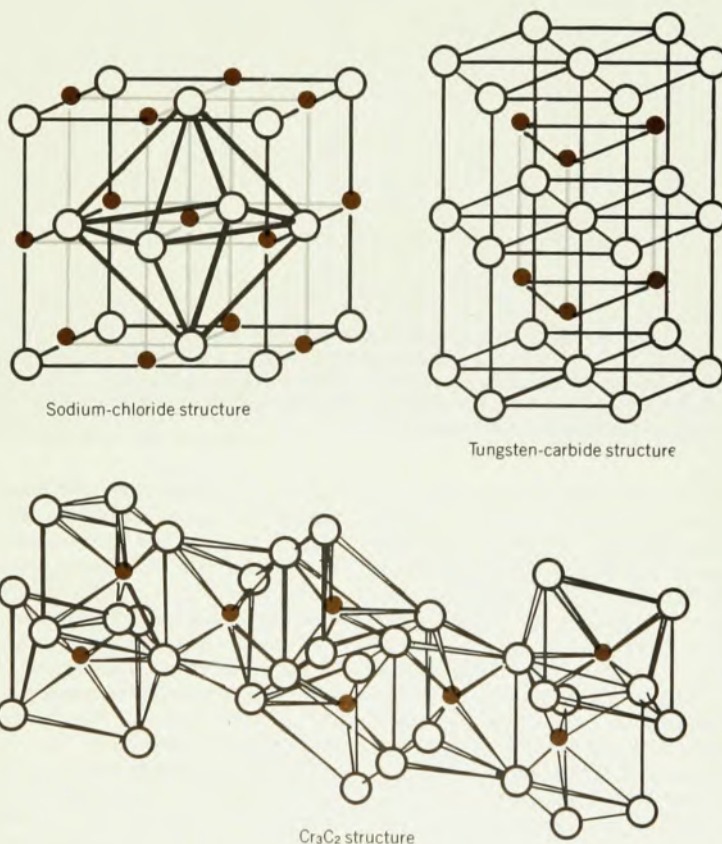


Physical properties depend on the non-metal to metal ratio and vacancy concentration; for example, in figure 3 we see that the melting temperature of the VC phase varies strongly across the composition range, with the maximum occurring at 46 atomic percent carbon. There is some disagreement as to where this maximum occurs, and in general the published transition-metal-metalloid phase diagrams containing the interstitial compounds as intermediate phases must be regarded as provisional.

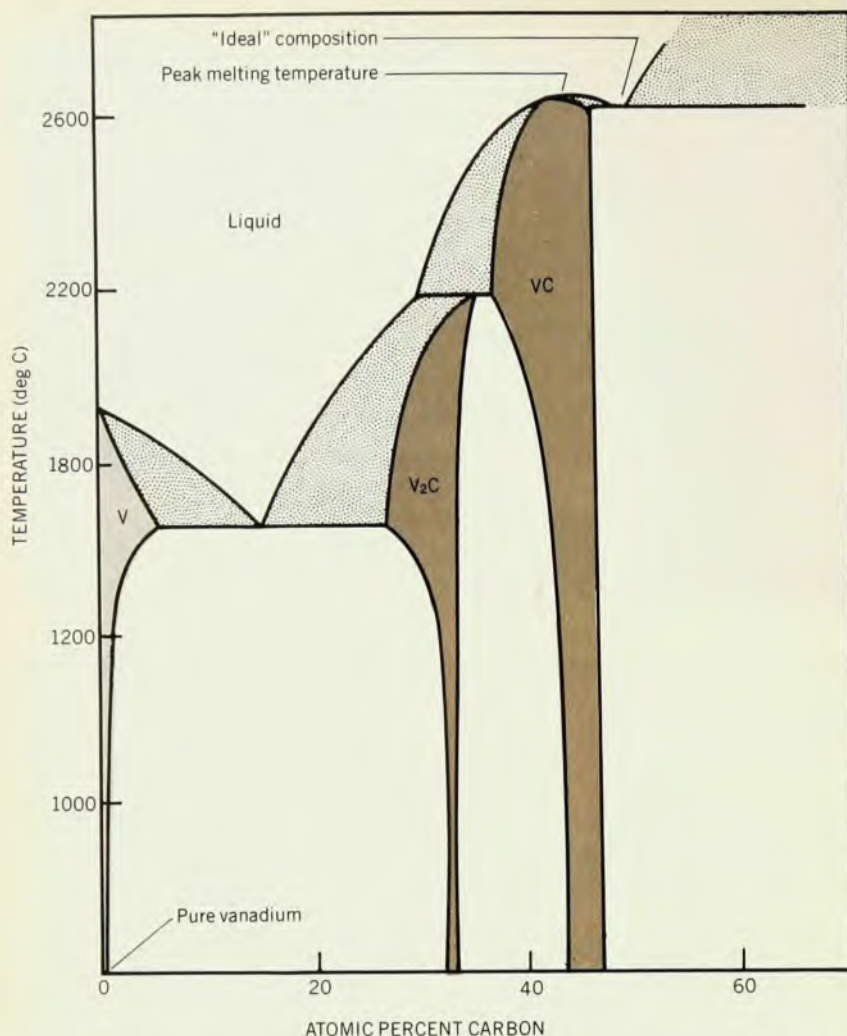
Resistivity, melting and hardness

In some of their properties—hardness, brittleness, high melting points—the interstitial compounds resemble ionically and covalently bonded solids, while in others—luster and conductivity—they appear to be good metals. Their room-temperature resistivities are typical of transition metals and are often lower than those of the pure metals from which they are derived. For example, the lowest reported room-temperature resistivity of ZrN is 7 microhm cm, while that of zirconium is 42; resistivities of the good conductors, aluminum and copper, are 2.6 and 1.7 microhm cm respectively. At high temperatures even the mechanical properties become metallic, for softness and malleability appear.

The interstitial compounds generally have high (often extraordinarily high) melting temperatures, usually higher than those of their parent metals. For example, titanium, zirconium and hafnium melt at temperatures from 2000 to 2500 K, whereas their monocarbides and diborides melt at temperatures 1000 to 2000 kelvins higher. Among the pure transition metals, the highest melting points occur in the Group VI column of the periodic table (chromium, molybdenum and tungsten). Among the monocarbides and



Structures of three interstitial compounds. Above are two simple Hagg compounds: the sodium-chloride structure, with metals (open circles) at fcc sites and metalloids (colored circles) at octahedral interstitial sites, and a coordination polyhedron illustrated with heavy lines; and the tungsten-carbide structure, with metals in simple hexagonal arrays and metalloids filling one of the two trigonal sites in the unit cell. Below is a complex, non-Hagg structure, a single unit cell of the Cr_3C_2 type most easily visualized with the coordination-polyhedron picture. Figure 2



Partial phase diagram of the V-C system, showing the composition ranges of the V₂C and VC phases. Solid and liquid are in equilibrium in the gray region, whose upper boundary is termed the "liquidus" and lower boundary the "solidus." Note the strong dependence of the solidus on metalloid-to-metal ratio, and that at the maximum of the solidus, melting is congruent—that is, solidus and liquidus coincide, as in a pure substance.

Figure 3

diborides, maximum stability tends to occur about Groups IV and V, while for the mononitrides, maximum stability appears to be a bit further left, at about groups III and IV. This behavior is illustrated in figure 4 for the 4d-series metals. As we shall see, such leftward shifts in the columns of the periodic table upon addition of metalloids occur for other properties as well. They have been correlated with trends in calculated band structures, and arise from the filling of, first, lower-lying bonding d states and subsequently of higher-energy antibonding states. The resulting picture suggests the notion of electron charge transfer from metalloid to metal; historically, this constituted the first battleground in the charge-transfer wars, of which more will be said later.

As noted above, the hardness of these materials is the property most frequently exploited commercially. Their use in cutting tools is well known, as is their

utility in the fabrication of wear-resistant parts—in dies, for example, and in hardening of steel. Their hardness appears to be directly related to bonding between the p electrons of the metalloid and the d electrons of the transition metal. Strong variations in hardness occur with changes in stoichiometry. The system TaC_{1-x} serves as a convenient example. As x ranges from 0 to 0.2, the hardness increases to a maximum, this variation being accompanied by a color change from golden when x is near zero to metallic white for x around 0.2. As with the trends in melting points, this phenomenon can be interpreted in terms of band-filling arguments. But interpretation and prediction of such properties and their variation is an area in which we have scant fundamental understanding, and to which physicists have only recently begun to contribute.

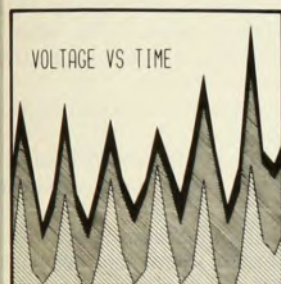
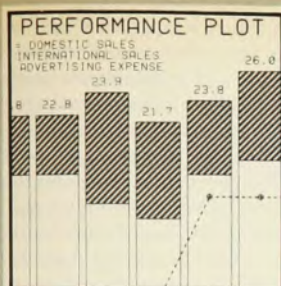
The correlation of band-theory predictions with the systematic variation in

several physical properties is illustrated for the 3d-diborides³ in figure 5. The density of states calculated for CrB₂ is shown at the bottom. Wave-function calculations show that below the broad minimum at about 1.1 rydbergs, the d-wave functions are bonding in nature and strongly hybridized with the metalloid p-electrons; above the minimum, the d functions are antibonding and only weakly hybridized. Given the band structure, the Fermi levels appropriate to the diborides of scandium, titanium and vanadium, as well as that of chromium, are indicated. The corresponding values of the melting points and low-temperature specific heats are plotted in the upper curves. A strong correlation between these properties and the electronic structure is evident. Band filling, and the consequent inclusion of more and more metallic character in the bonding, serves as a sort of "Matthias connection," relating disparate properties in a rational way.

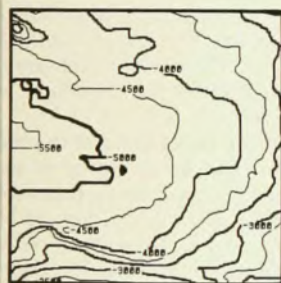
Superconductivity

Interstitial compounds of the rock-salt structure include superconductors whose transition temperatures, T_c , are exceeded only by those of the materials with A15 structure, such as Nb₃Sn. Figure 6 shows the T_c 's of a few of the better performers, the niobium carbides and nitrides, as a function of metalloid content. The highest known T_c for an interstitial compound, 17.5 K, occurs for the mixed carbide-nitride NbC_{0.3}N_{0.7}. The best performers of several other classes of superconductors, pure metals, A15's, and so on, are also shown for comparison. Bear in mind that the interstitial compounds are difficult to prepare, in part because of their high melting points, and they are difficult to characterize, in part because of their inclination to lie off stoichiometry. They also suffer from interfering effects of oxygen and other impurities; T_c 's higher than those presently known may in fact occur. Ted Geballe (University of Washington) has presented an interesting view of the superconductivity of NbN in which the role of the niobium d electrons is downplayed. He notes that this compound has low-temperature properties similar to those of lead, suggesting that this compound, with a lattice greatly stiffened by the presence of the interstitial metalloid, is simply "stiff lead." Structure, of course, plays a role in T_c ; for example, in contrast to the metastable rock-salt structure form of tungsten carbide for which T_c is 10.0 K, the hexagonal phase has a T_c of only 2.5 K.

The addition of interstitial hydrogen to Pd, Pd-Ag and Pd-Cu produces⁴ superconductors of moderately high T_c . These materials are unusual in that none of the host metals are superconductors. Detailed and gratifyingly accurate theoretical predictions of their superconducting properties, including the reverse isotope



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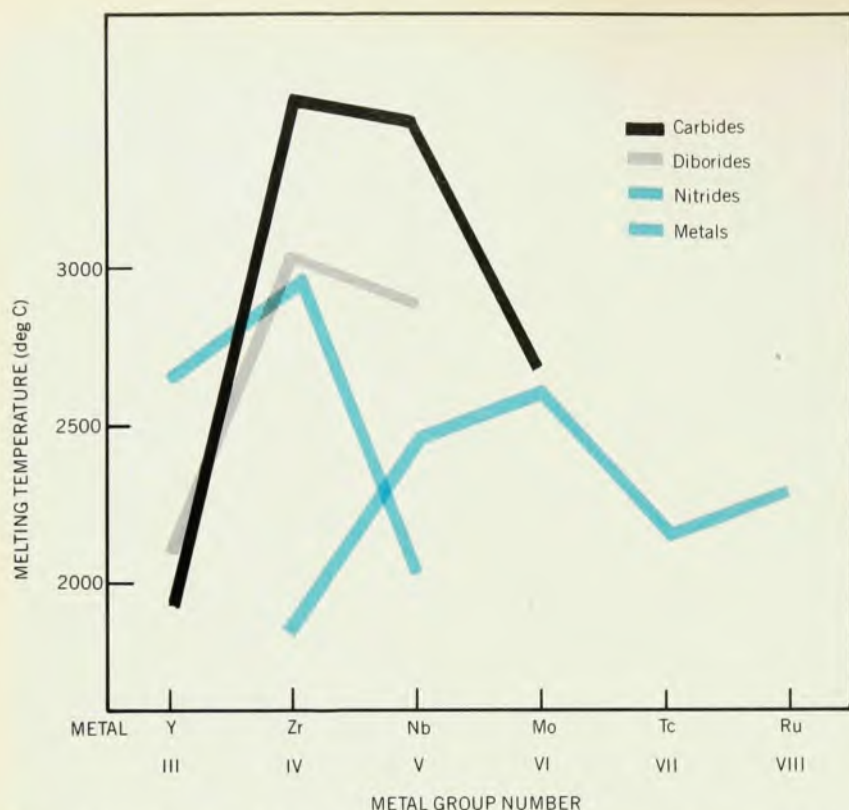
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Variation of the melting point of the 4d metals and their carbides, nitrides and diborides with metal group number. Note the leftward shift upon addition of metalloids. Figure 4

effect, have been carried out by Dimitri Papaconstantopoulos (George Mason University), Barry Klein (Naval Research Lab), and Al Switendick (Sandia). This phase of the study of interstitial alloys differs from others in that much of the work has been done by physicists rather than metallurgists.

Hydride formation

There are other interesting aspects of hydride formation. In the regime of dilute hydrogen concentration, the hydrogen atoms interact via strain fields, which are of very long range. Properties become a function of the shape of the host, and strictly classical theories are applicable.⁵ Also, in pressure-concentration phase diagrams, the critical points atop immiscibility regions occur at experimentally accessible pressures, making the hydrides of interest to those concerned with phase transitions and critical phenomena. As

for practical matters where the metal-hydrogen problem is of great concern, these range from the embrittlement associated with the introduction of hydrogen in steels to questions of hydrogen storage. One can store more hydrogen per unit volume in a metal host than in a high-pressure tank; the problem is to find a cheap, light host to which hydrogen can be added and removed at reasonable pressures and temperatures.⁶

Current champion hydrogen storers are based on either LaNi_5 or TiFe , which hold up to about 6 and 2 hydrogen atoms per formula unit at room temperature and pressures of a few atmospheres. The former is not very cheap (although costs have been sharply reduced of late), and neither is very light. Still, either can be used in light canisters to reduce the volume of hydrogen storage on the forklift truck of figure 1 by a factor of three, with some weight savings as well. Magnesium

hydride could be employed where weight is a critical factor, but inconveniently high temperatures and pressures are needed to store and deliver hydrogen with this medium. Introducing transition elements may improve the magnesium-based hydrides. There has been considerable effort involving electron energy band and semiempirical theories of these hydriding problems. Dan Gelatt and coworkers at Harvard have, for example, estimated the heats of formation of the 3d and 4d transition-metal hydrides. The effort involved sorting out the various contributing factors which are:

- ▶ the formation of metal-hydrogen bonding band,
- ▶ the lowering of the metal d bands due to the presence of the positively charged protons, and
- ▶ the band filling associated with the addition of the extra electrons contributed by the hydrogens.

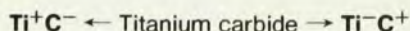
Magnetic properties and catalysis

An example of the magnetic properties of the interstitial compounds appears in figure 7 where the magnetic moments of the semi- and monoborides and of the host metals are plotted. Both the moments and the Curie temperatures of the borides are comparable to those of the pure metals. Note the similarity of the boride and metal curves. Again, as with melting temperatures, we see a shift to the left in the periodic table when half, and then in turn one, boron atom is added per metal atom. These compounds are good hard magnets; that is, they have large coercive forces.

Borides and phosphides of the transition metals can be produced in amorphous form. Some of the earliest work on amorphous materials was done at NBS, and among the first to be developed were "electroless" nickel and cobalt. These are metal-rich phosphides that provide bright, hard plating by autocatalytic deposition. By catalysing its own reaction this process is less energy consuming than is electroplating. As deposited, these platings are amorphous. Heat treating at 350°C or above produces ordered, harder coatings, with lowered corrosion resistance. This class of plating is widely used in industry today.

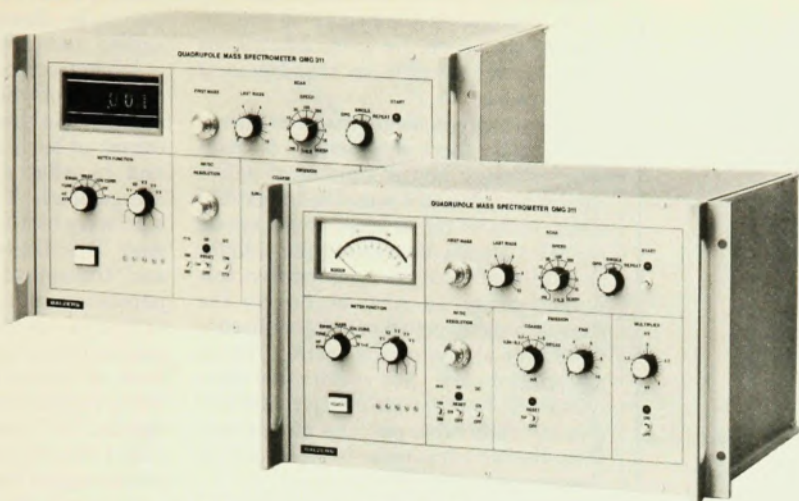
A number of these amorphous materials have interesting magnetic properties. Some are ferromagnetics, others anti-ferromagnetics; transition-metal borophosphides have been commercially developed to be used for magnetic shielding. These compounds also have tensile strengths and electrical properties similar to steels. They are produced continuously by rapid freezing from a melt, resulting in long ribbons of material. The process is economically competitive and such material might, for example, be used in automotive tires. The devitrification temperature of the amorphous interstitial compounds is typically one-half the

Charge transfer: a case history



Photoelectron spectroscopy
X-ray transitions
Band theory
Compton scattering
Electronegativity arguments
Engel-Brewer theory

Photoelectron spectroscopy
X-ray transitions
Band theory
The "leftward shift" of properties (as seen in figures 4 and 7)



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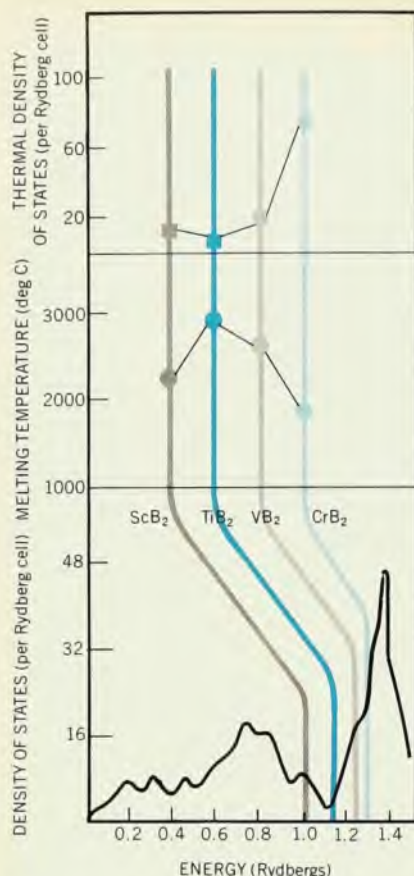
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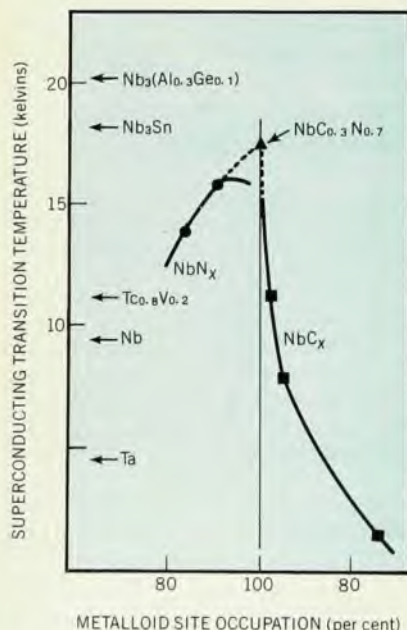
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Correlation of the melting point and low-temperature specific heat of the 3d metal diborides with filling of the d bands. Figure 5



Variation of the superconducting transition temperature of the carbides and nitrides of niobium with metalloid content. The transition temperatures of a mixed compound and several pure metals and alloys are shown for comparison at the left of the graph. Figure 6

melting temperature. Just as the properties of the crystalline compounds depend strongly on the degree of stoichiometry, the properties of the amorphous ones depend strongly on the degree of lattice order.

Where there is magnetism and superconductivity there also tends to be catalytic activity,⁷ and a number of active catalysts are found among the interstitial compounds, WC and Mo₂C for example, and also among the related phosphides and sulfides. We have argued that this property may, in part, be due to the change in the structure of metal sublattice from that of the parent metal. These materials do not yield remarkable catalytic rates, but they are cheap relative to platinum and other precious metals, and they display advantages in resistance to poisons and corrosive reactants.

As a general rule, the interstitial compounds have lower work functions than the transition metals. Consequently there are many strong thermionic emitters among them, NbC, LaB₆, ZrB₂ and TiC being notable performers. This technologically important property is not well understood in terms of current theory, and much work remains to be done in this area. Strong thermionic emission, of course, coupled with high melting point and resistance to corrosion and erosion, marks these materials as likely candidates for use as electrodes in magnetohydrodynamic channels.

Bonding

Not surprisingly, the dichotomy of the physical properties spills over into thinking on the electronic structure. The fact that the molecular volume of, say, titanium carbide is significantly smaller than the sum of the elemental atomic volumes of titanium and carbon implies a significant amount of covalent bonding in these materials. The mechanical properties and the occurrence of compounds are commonly accepted as indicative of strong covalent metalloid-metal p-d electron bonding, while the occurrence of transition-metal-like electronic properties and the existence of wide stoichiometric ranges suggest the importance of metallic bonding among the transition-metal d electrons. The d-d metallic bonding is modified when a compound is formed because the metal-metal distances become somewhat larger and, more importantly, because the symmetry of the metal sublattice is different from that of the parent metal. To a first approximation, the electronic density of states seen in figure 5 is associated with the transition-metal d electrons and depends critically on the symmetry of the metal sublattice. There is, however, substantial metalloid electron admixture into these states, and the extent to which the bands are filled and in turn the electronic properties of the compounds are affected by this ad-

mixture. While the interplay of p-d covalency and of d-d metallic behavior has yet to be disentangled, it is generally agreed that they are both significant.

In contrast, controversy surrounds the questions of whether or not there is ionic character in these materials, of what the extent of such ionic bonding might be, and in what direction the associated charge flows. Titanium carbide has been a primary battleground, and the table on page 38 indicates classes of experiment and theoretical argument involved in papers maintaining that there is evidence of flow in one direction or the other. (A similar listing for table salt would put almost all entries in the Na⁺Cl⁻ column.) Some papers conclude that there is substantial Ti-C charging, say of the order of a half electron charge per site, whereas others suggest that it is small, less than 0.1 e in magnitude. In view of the metallic character of these materials and the attendant screening this allows, the smaller limit is more plausible physically. For that matter, few of the arguments for the larger charge are convincing in themselves. Charging as small as 0.05–0.1 e is, nevertheless, physically significant in most models into which it might be inserted, as into an electrostatic (Madelung) contribution to the cohesive energy.

Not all the entries in the table are correct in their arguments, in our opinion, but there are at least a few in each column that have taken a physically sensible model and correctly used the data at hand to obtain their results. In these cases, charge flow has become a parameter rather than an absolute physical quantity. An absolute definition of charge flow from one atomic site to another requires a definition of the sites—for example, assignments of their volumes.⁸ There has yet to be satisfactory agreement on this issue. The problem is difficult for metals and particularly so for the interstitial compounds with their interplay of metallic, covalent and, possibly, ionic character and remains a major challenge for the solid-state scientist. In contrast, significant progress has been made on the "simpler" problem of bonding and charge flow in semiconductors by Linus Pauling, William Pearson, James Phillips and others.

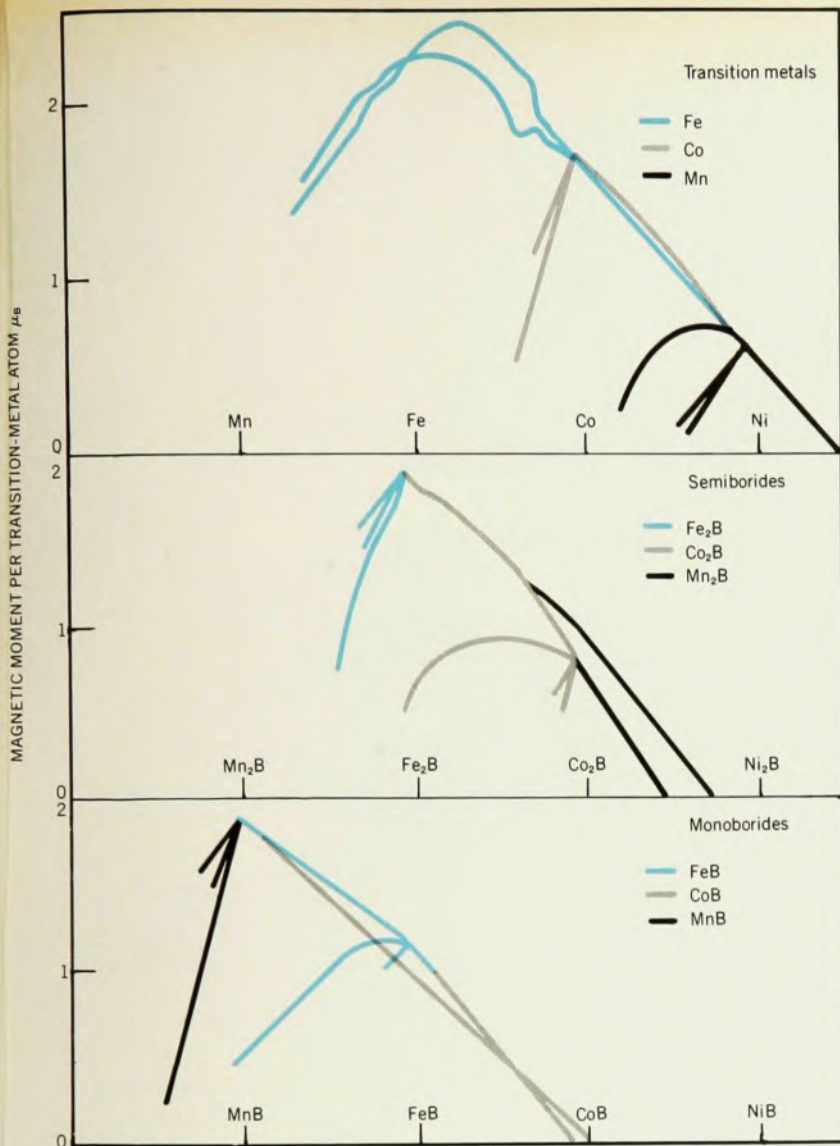
There has been a half century of work on interstitial compounds by crystal chemists and metallurgists; much of the resulting literature is relevant to the physicist today, who can find challenging problems and fascinating possibilities for technology in this field.

* * *

This article is an adaptation of an invited talk given in March 1976 in Atlanta, Ga., at the Solid State Meeting of The American Physical Society.

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The magnetic moment of the 3d metals and their alloys, and of the semiborides and borides of the metals and alloys, shown schematically. Fe, Co, Mn and Ni-based alloys are represented in color, gray and black as shown. For full details and sources of the data represented by these curves, see M. C. Cadeville and E. Daniel, *J. Physique* **27**, 499 (1966). Figure 7

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