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STUDY OF MATERIALS HAVING SIGNIFICANCE FOR HIGH ENERGY

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CHEMISTRY W E WALLACE 15 JUL 83 ARO-17165.42-MS

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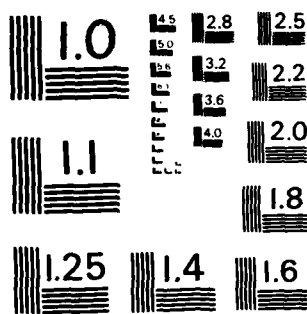
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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
REPORT NUMBER FINAL REPORT 17165.42-MS	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
TITLE (and Subtitle) Study of Materials Having Significance for High Energy Magnet Production and for Hydrogen Storage		5. TYPE OF REPORT & PERIOD COVERED FINAL 7/1/80 - 6/30/83
AUTHOR(s) W. E. Wallace		6. PERFORMING ORG. REPORT NUMBER
PERFORMING ORGANIZATION NAME AND ADDRESS Department of Chemistry University of Pittsburgh Pittsburgh, PA 15260		8. CONTRACT OR GRANT NUMBER(s) DAAG29-80-K-0036
CONTROLLING OFFICE NAME AND ADDRESS U. S. Army Research Office Post Office Box 12211 Research Triangle Park, NC 27709		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS P-17165-MS
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE 7/15/83
		13. NUMBER OF PAGES 14
		15. SECURITY CLASS. (of this report) unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) JVB		
18. SUPPLEMENTARY NOTES THE VIEW, OPINIONS, AND/OR FINDINGS CONTAINED IN THIS REPORT ARE THOSE OF THE AUTHOR(S) AND NOT NECESSARILY OF THE GOVT. STATED AS AN OFFICIAL DEPARTMENT OF THE ARMY POLICY OR DECISION, UNLESS SO DESIGNATED BY OTHER DOCUMENTATION.		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Magnetism, rare earth alloys. Magnetocrystalline anisotropy. $\text{Sm}_2\text{Co}_{17-x}\text{Zr}_x$, Magnetism and Crystallography. $\text{Co}_2\text{Co}_{17-x}\text{Zr}_x$, Magnetism and Crystallography. Zr doping, effects on Anisotropy. ErPrCo_{17} Magnetism. TmPrCo_{17} Magnetism. YbPrCo_{17} Magnetism. Rare Earth inter-metallic hydrides. ZrCo_x Alloys, Magnetism. Hydrogen Absorption. Magnetism of hydrides. Kinetics of hydrogen absorption.		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The work carried out between July 1, 1980 and June 30, 1983 is described in detail in 6 review articles and 38 articles providing accounts of original work. The distribution of published papers is as follows: Journal of Magnetism and Magnetic Materials - 10, Solid State Communications - 6, Journal of the Less-Common Metals - 5, Journal of Applied Physics - 4, Journal of Solid State Chemistry - 4, Journal of Physical Chemistry - 3, Journal of Physics and Chemistry of Solids - 1, and chapters in various books - 11. A considerable part of the work has been concerned with "the Zr Effect," the		

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beneficiation of permanent magnet properties of rare earth-cobalt systems by doping with small amounts (≈ 1 wt%) of Zr (or Ti or Hf). It has been found that Zr replaces Co in R_2Co_{17} systems, and this results in a sharp increase in magnetocrystalline anisotropy. This is responsible for part (but undoubtedly not all) of the Zr effect.

Systems such as $ErPrCo_{17}$ are found to exhibit enhanced magnetocrystalline anisotropy, compared to Er_2Co_{17} or Pr_2Co_{17} , since Er and Pr are observed to preferentially occupy the 2b and 2d sites, respectively. In these sites each has uniaxial anisotropy, as predicted from single ion anisotropy theory. Preferential site occupancy occurs because of the size difference between Er and Pr. Similar effects are observed in $RPrCo_{17}$ systems with $R = Tm$ or Yb .

Ce in $CePd_3$ is in a mixed valent state. Si can be introduced in the lattice to form $CePd_3Si_x$. This ternary contains Ce in the Ce^{3+} state.

Studies of RCo_2-xFe_x hydrides show that whereas the R and Co moments are decreased by hydrogenation, the Fe moment is unchanged. In Y_6Fe_{23} the Fe moment is actually raised about 30% by hydrogenation, to $2.6 \mu_B$. The results suggest that H removes electrons from the Fe d-band.

The work to date indicates that surface features are highly significant for materials involved in hydrogen storage and for those materials involved in the fabrication of high energy magnets. It appears likely that surface Co is the nucleation site for inverse domains and may hence control coercive force.

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Study of Materials Having Significance for High Energy Magnet Production
and for Hydrogen Storage

by

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FINAL REPORT

July 15, 1983

to

U.S. Army Research Office

Contract No.: DAAG29-80-K-0036

Period Covered: July 1, 1980 - June 30, 1983

APPROVED FOR PUBLIC RELEASE:

DISTRIBUTION UNLIMITED

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I. INTRODUCTION

The work for the period July 1, 1980 to June 30, 1983 falls into two general categories: (1) Study of the Magnetic Properties of Intermetallic Compounds and (2) The Study of Metallic Hydrides. The completed work is described in detail in the 44 publications listed in Section VI below. Since all the significant accomplishments in the work have appeared in the open literature, the present report is prepared with two objectives in mind: (1) to indicate where the work is published (see Section VI) so that the reader can gain access to it with a minimum of difficulty and (2) to highlight a few of the completed works which appear to be of more than ordinary interest. In Section II a brief account is given of the circumstances which led the author to prepare the 6 review papers listed in Section VI-A. Selected studies pertaining to Magnetism of Intermetallic Compounds, Magnetism of Hydrides of Intermetallic Compounds and Surface Chemistry as it relates to magnetism and hydride formation are discussed in the subsequent Sections III, IV and V, respectively.

II. REVIEW PAPERS

It seems appropriate to indicate the circumstances which led to the six review papers given in Section VI below. The author was selected as the recipient of the first Frank H. Spedding Award for excellence in Rare Earth research. (1)* was the Spedding Award Address. (2) was an invited address at a conference on Solid State Chemistry. (3) and (8) formed part of the first binational U.S.-India Conference on Rare Earth Materials, held in Cochin, India

*The numbers in parenthesis refer to the publications listed in Section VI.

in 1980. The author was Chief-of-Party of a contingent of 10 Americans who took part in that conference. (4) was an invited lecture which was presented at the 14th Rare Earth Research Conference. A NATO Advanced Study Institute was held in Rhodes, Greece in 1980. (5) is the written version of two invited lectures given at that Institute.

Collectively, these reviews provide summary accounts of (1) the magnetism of the industrially important R_2Co_{17} systems, (2) the mechanism of hydride formation in hydrogen storage materials and (3) the effects of hydrogenation on the electronic properties of metallic systems, revealed primarily by magnetic measurements.

III. MAGNETISM OF INTERMETALLIC COMPOUNDS

A. Rare Earth Systems - Enhancing Anisotropy

1. The Zr Effect

It is well known that additions of small amounts of Zr (~ 1 wt%) has a remarkable effect in improving the practical magnetic properties - energy products and coercive forces - of Sm_2Co_{17} -based alloys. This is also the case for Ti and Hf additions. For simplicity, this influence of Zr, Ti or Hf doping on permanent magnet properties is called "The Zr Effect." The origin of this effect is at present obscure. It has been partially elucidated in publications numbers 16,17,19 and 26 in Section VI.

A necessary, but not sufficient, condition for a large energy product is a strong magnetocrystalline anisotropy. $SmCo_5$ is exceptional in this respect. Its anisotropy field (H_A) is roughly 400 kOe. In contrast, Sm_2Co_{17} exhibits an H_A of about 50 kOe, which is small for practical applications. One possibility for the occurrence of the Zr Effect is that Zr enters the lattice and sharply increases H_A . The several studies mentioned in the preceding paragraph were initiated to examine this possibility.

Earlier work* had shown that H_A is increased in R_2Co_{17} systems by the substitution of Co in these materials by Fe, Mn, Cr, etc. It seemed possible, therefore, that Zr, etc., would have a similar but larger effect. Therefore R_2Co_{17} systems (with $R = Ce, Sm, Er$ and Yb) were doped with V, Ti, Zr and Hf and their magnetic properties examined. Initially it was not clear where these elements would substitute - for R or for Co. A variety of evidence indicated that Zr, etc., substituted for Co. The strongest evidence was crystallographic in nature. The unit cell increased in size with increasing doping. This is expected if Zr replaces Co because of the relative sizes involved. If R were replaced by Zr, a smaller atom, a shrinkage in unit cell size would be anticipated.

Actually, there was an even earlier question: Would Ti, Zr, etc., enter the R_2Co_{17} lattice? It was not clear at the outset of this work that this would occur. Perhaps they would separate out entirely. Again, crystallographic evidence provided decisive information. For example, it was found that whereas Sm_2Co_{17} has the rhombohedral Th_2Zn_{17} structure, $Sm_2Co_{16}Zr$ has the hexagonal Th_2Ni_{17} structure. This structure change clearly indicates that Zr is entering the lattice.

Experiment showed that Zr is 2 to 3 times more effective in enhancing H_A than Fe, Mn or Cr. The substituent effect in $R_2Co_{17-x}T_x$ alloys is in the order $T = Zr > V > Hf > Ti > Cr > Mn > Fe > Cu$. It thus appears that a part, but probably not all, of the Zr Effect is the strengthening of the magnetocrystalline anisotropy of the system. Presumably this occurs because Zr preferentially substitutes at the Co dumbbell sites, removing the adverse effect of this

*See, for example, H. J. Schaller, R. S. Craig and W. E. Wallace, "Magnetic Characteristics of Some Binary and Ternary 2-17 Compounds," J. Appl. Phys. 43, 3161 (1972). See also W. E. Wallace and K. S. V. L. Narasimhan, "Magnetic Properties of 2:17 Rare Earth Systems," in The Science and Technology of Rare Earth Materials, W. E. Wallace and E. C. Subbarao, eds., Academic Press, Inc. New York (1980), p. 329.

kind of Co on H_A . This, at present, is merely a postulate which needs to be subjected to experimental test.

2. Increasing H_A by Rare Earth Substitution

In a material such as Er_2Co_{17} (hexagonal, Th_2Ni_{17} structure) there are two crystallographically distinguishable types of rare earth. In Wyckoff notation these are atoms (or ions) in the 2b and 2d sites. Single ion anisotropy theory shows that the interaction is always opposite at these two sites. If R at the 2b site exhibits uniaxial anisotropy, then R at the 2d site exhibits planar anisotropy; and vice versa. If one could form a system $R'R''Co_{17}$ in which R' and R'' occupied the 2b and 2d sites preferentially, and if these rare earths were chosen so that the interactions in both cases were uniaxial, then H_A would be substantially increased over that for R'_2Co_{17} or R''_2Co_{17} . This follows because in the binary systems there is always competition between the rare earths in the 2b and 2d sites, and this competition keeps H_A artificially low.

$ErPrCo_{17}$ is a favorable system if Er and Pr occupy the 2b and 2d sites, respectively. ^{166}Er Mössbauer spectroscopy indicates (14) that the desired site occupancy does (partly) occur. Replacement of 20% of Er by Pr in Er_2Co_{17} leads to a 40% increase in the room temperature value of H_A (paper 14 in Section VI). Similar effects were observed in Tm_2Co_{17} - and Yb_2Co_{17} -based systems. These systems are discussed in papers (8), (12), (20) and (26) in the list in Section VI.

B. Systems Exhibiting Miscellaneous Effects

In paper (22) it is shown that a valence change for Ce in $CePd_3$ can be induced by introducing Si to form the ternary alloy $CePd_3Si_x$, where $0 \leq x \leq 0.3$. Ce in $CePd_3$ is in a mixed valence state, as indicated by its crystallographic features and also by its susceptibility. With Si introduction, both of these

measurements indicate a transformation into Ce^{3+} . Expansion of the lattice by Si entry enlarges the space available for Ce in the lattice and favors the formation of the larger tripositive ion.

Co forms many Laves phase compounds. These can be represented as ACo_2 . The ACo_2 systems are exchange-enhanced Pauli paramagnets if A is non-magnetic, e.g., YCo_2 , and are ferromagnetic (or ferrimagnetic) if A is magnetic, e.g., $PrCo_2$. $ZrCo_2$ is a paramagnetic material. It was found (11) that $ZrCo_2$ can be made hyperstoichiometric in Co, and this non-stoichiometric material becomes ferromagnetic at low temperatures. Magnetic ordering occurs only for $ZrCo_x$ where $x \geq 2.8$. Curie temperatures reach 160°K in this system.

Th_6Mn_{23} and Y_6Mn_{23} are isostructural. They are face-centered cubic materials with 116 atoms per unit cell. Th_6Mn_{23} is a Pauli paramagnet; Y_6Mn_{23} is a ferrimagnet with $T_c = 480^\circ K$. The ordered magnetism in Y_6Mn_{23} is quenched by hydrogenation, whereas hydrogenation induces magnetic order in Th_6Mn_{23} . Given these contrasting features, it was of interest to ascertain the behavior of $Th_{6-x}Y_xMn_{23}$ ternary alloys. These ternaries exist in the Th_6Mn_{23} structure for all values of x, from 0 to 6.

The ternaries are found to be magnetically ordered for $1.5 \leq x \leq 6$. T_c steadily increases as the Y content of the ternary alloys is increased. Th_5YMn_{23} exhibits only short-range magnetic order.

IV. HYDROGENATION AND ITS EFFECT ON MAGNETISM

In studies of $RCo_{2-x}Fe_x$ hydrides (37) it was found that hydrogenation simultaneously decreases T_c , the cobalt moment and the R-Co and R-Fe exchange interactions. In contrast, there is little change in the iron moment by hydrogenation. The effect of hydrogenation on iron moments in rare earth-iron systems is further alluded to below, where an actual increase in Fe moment is observed.

In studies (34) of ZrMn_2 and its hydrides it has been observed that ZrMn_2 is a Pauli paramagnet ($x = 1.6 \times 10^{-3}$ emu/formula unit at 295°K), whereas $\text{ZrMn}_2\text{H}_{3.6}$ is a weak ferromagnet with $T_c = 143^\circ\text{K}$. The work seems to show that hydrogen extracts electrons from the d-band. This and the enlargement of the lattice transforms the host metal from a Pauli paramagnet into an ordered magnetic material.

Rare earth intermetallics of the formula R_6Fe_{23} are of considerable interest. At one time it was thought that they, being predominantly Fe alloys, would be economical hydrogen host materials. It now appears that the hydrides they form are too stable to be used for hydrogen storage. Even so, they are very interesting scientifically and they have been thoroughly examined in this laboratory. Interest has centered on the location of hydrogen and the influence of hydrogen on magnetic behavior.

In (33) it was found that (cubic) $\text{Er}_6\text{Fe}_{23}$ becomes tetragonal when hydrogenated to $\text{Er}_6\text{Fe}_{23}\text{H}_{14}$. When further enriched in hydrogen, the material becomes cubic again.* Examination of these material by neutron diffraction (42) indicates why these crystallographic changes occur. There are three equivalent sets of sites in planes perpendicular to x, y and z axes. At $\text{H}/\text{Er}_6\text{Fe}_{23} = 14$ two of these sets of sites are occupied by hydrogen, whereas one set is empty. This asymmetry leads to the tetragonal distortion.* When further hydrogenated, the other set of sites is populated and cubic symmetry is restored.

In studies of Y_6Fe_{23} and its hydride, it was observed** that the magnetization (in $\mu_B/\text{f.u.}$) of Y_6Fe_{23} and $\text{Y}_6\text{Fe}_{23}\text{H}_{16}$ are 45.4 and 59.8, respectively. This indicates Fe moments of 2.0 and 2.6 μ_B/atom in the parent alloy and

* H. Kevin Smith, Ph.D. Dissertation, University of Pittsburgh (1983).

**A. T. Pedziwiatr, E. B. Boltich, W. E. Wallace and R. S. Craig, in Electronic Structure and Properties of Hydrogen in Metals, P. Jena and C. B. Satterthwaite, eds., Plenum Publishing (1983), p. 367.

hydride, respectively. The reduced moment in the parent alloy, compared to elemental Fe, is ascribed to electron transfer from Y to Fe, resulting in a more nearly filled d-band. Similar reasoning suggests that H removes electrons from the d-band during hydrogenation. The large Fe moment is one of the largest, if not the largest, Fe moment observed in metallic systems.

V. SURFACE CHEMISTRY, MAGNETISM AND HYDRIDE FORMATION

The model developed several years ago for the hydrogenation of LaNi_5 is as follows:

1. LaNi_5 oxidizes to give $\text{La}_2\text{O}_3 + \text{Ni}$ at the surface.
2. H_2 adsorbs dissociatively on the surface Ni.
3. Monatomic hydrogen penetrates the oxidized surface layers and reaches the underlying bulk.
4. Diffusion of H in LaNi_5 occurs.
5. As the H content builds up, the rhombohedral β phase forms.
6. Step 3 is rate-controlling.

The presence of elemental d-transition metal is essential for rapid hydrogenation kinetics.

Similar surface features undoubtedly exist for RCo_5 and R_2Co_{17} systems which are significant as permanent magnet materials. Undoubtedly, R_2O_3 and Co exist in the surface. Co is magnetically soft and may be the site for nucleation of inverse domains. The surface cobalt may control coercivity. It in turn is generated by oxidation.

From these remarks, it is clear that the surface chemistry provides a conceptual link between studies of hydrogen storage materials and high energy

*W. E. Wallace, R. F. Karliceck, Jr. and H. Imamura, J. Phys. Chem. 83, 1708 (1979).

magnet materials. Surface oxidation is deleterious in the second case but not in the first. The present studies focus attention on the importance of establishing surface chemistry in the development of new, technologically significant magnetic materials.

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