

stability and disruption behaviour provoked by the α heat source.

The most important diagnostics in this phase will undoubtedly be the neutron diagnostics, but other systems which are essential in unravelling the energy balance, such as the bolometers will retain a key importance as will diagnostics determining impurity and instability behaviour, such as the spectroscopic and X-ray systems. The wall diagnostics will be required to give information on the plasma wall and radiation damage behaviour in this phase. It would also be useful to have direct information on α particle energy transfer and containment but it is not clear at present that suitable diagnostic techniques can be developed to operate in the intense radiation field. Information on the effectiveness of the α particle heating will however be obtained from the neutron measurements. The various systems giving spatially resolved information on temperature and density profiles will be essential for detailed energy balance analysis.

Time Scales

The start of Phase I in mid-1983 depends, of course, on the present construction schedule being maintained. The beginning of Phase II, shown as 1984, however, depends on the availability of additional heating and diagnostic equipment which in principle is not linked to the main construction programme. Thus Phase II is subject to delays of its own but is not sequentially dependent on Phase I, which if necessary could be curtailed. It would be possible and indeed preferable to begin directly with additional heating and a full set of diagnostics if these were available. Consequently, it seems reasonable to suppose that some form of Phase II operation will take place during 1984.

The beginning of Phase III operation shown as 1985 depends on the availability of advanced high power heating equipment. This in turn depends on the solution of design and construction problems. The total time allocated for Phases II and III in Table 1 is 3½ years. In very favourable circumstances and given a full year for Phase I this time might be reduced to 2½ years. Of course if impurity, instability or reliability problems prove serious in the earlier phases, this date will be delayed and, in the worst case, D-T operation will not be undertaken in JET at all. Nevertheless, the date of 1988 shown in the Table for commencement of D-T operation appears to be reasonable, although it does assume a certain element of good fortune backed by sound planning, timely resource commitment and forceful management.

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Hydrogen in Intermetallic Compounds

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Intermetallics are single phase stoichiometric compounds of two or more elemental metals. Many of them react readily with gaseous hydrogen and form exothermally, hydrides of convenient stability which can be used for reversible H_2 storage. The high density of the stored H, the good kinetics of the H absorption and desorption, as well as safety aspects, make H_2 storage by intermetallics very attractive and are the main reasons for the rapid growth of applied research in this field over the last ten years. The related phase transitions, structure transformations, variations of the electronic and magnetic properties of bulk and surface, phonon properties of the metal lattice and of the interstitial H and H diffusion represent a broad and exciting field for basic research in solid state and material science.

The actual research is concentrated on compounds of the following four groups:

- AB_2 compounds (e.g. $ZrMn_2$, $TiMn_{2-x}$, so called Laves phases)
- AB_5 compounds (e.g. $LaNi_5$, which is the "drosophila" of the H_2 storage compounds)
- FeTi based compounds
- Mg based compounds (e.g. Mg_2Ni , La_2Mg_{17} , alloys of Mg)

As is evident from these examples, at least one of the components is a hydride former (Zr, Rare Earth, Ti, Mg, Pd, ...).

Whereas many physical and chemical properties of hydrides of intermetallic compounds (ternary hydrides) are comparable with those of hydrides of elemental metals (binary hydrides), there are also some pronounced differences:

- intermetallics are, by far, more reactive to H_2 and less sensitive to impurities in the gas;
- the brittle intermetallics disintegrate into powder;
- H-bonding is weaker and can be adjusted by substitutions or through off-stoichiometric mixtures.

Phase Diagrams

The H absorption behaviour can be described in terms of pressure-composition-isotherms (Fig. 1). The intermetallics are able to dissolve some atomic percent H in the α phase, but then as the H concentration is increased, the H-H interaction becomes locally important and nucleation and growth of the hydride phase (β) start. Whilst the two phases ($\alpha + \beta$) co-exist, the isotherms show a plateau, the length of

which determines how much H_2 can be stored reversibly with small pressure variations. In the pure β phase, the H_2 pressure rises steeply with the concentration. At higher H_2 pressure further plateaus and further hydride phases (γ , σ , ...) may be found. The two phase region ends in a critical point T_c .

For $LaNi_5$ the first plateau is at 1.6 bar at room temperature and the α and β phase correspond to $LaNi_5H_{0.4}$ and $LaNi_5H_6$, respectively. A further plateau exists above 1 kbar corresponding to the formation of $LaNi_5H_9$.

Crystal Structure

From neutron and X-ray diffraction as well as from the shape of the pressure-composition isotherms, it is known that the dissolved H goes on tetrahedral or octahedral interstitial sites of the metal lattice. Lattice expansions of ≈ 20 vol % between the hydride phase (β) and the solid solution phase (α) are normal. As a result, the brittle intermetallics disintegrate into powder when they go for the first time through the two phase region. After a few absorption / desorption cycles powder grains smaller than $10 \mu m$ with many cracks and defects are created (Fig. 2), yielding a large specific surface of $\approx 1 m^2/g$.

The formation of a high density of lattice defects with the H absorption leads to an anisotropic broadening of the diffraction lines which makes the unambiguous indexing of the powder pattern difficult, particularly in the case of $LaNi_5H_6$, for which two crystallographically different structure models (P31m and P6/mm) have been proposed.

At room temperature, the H atoms are randomly distributed among the available

Fig. 1 — Pressure-composition isotherms and phase diagram of a metal-hydrogen system.

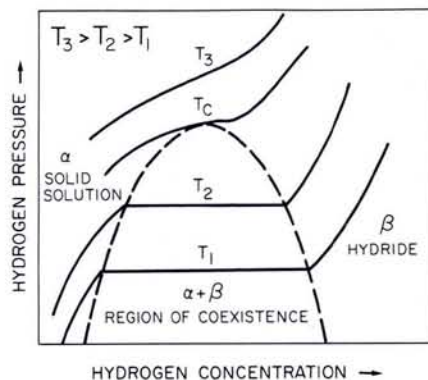




Fig. 2 — Scanning electron micrographs of LaNi_5 after a few hydrogen absorption/desorption cycles.

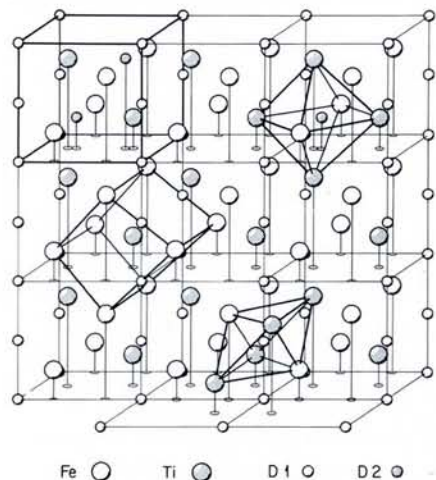
interstitial sites of that phase. Ordering occurs at low temperature.

The occupation of different interstitial sites does not necessarily lead to different hydride phases with different plateau pressure. In the β phase of FeTiH_x , for example, H atoms occupy simultaneously two different octahedral sites (the sites D1 and D2 in Fig. 3), both with 2 Fe and 4 Ti atoms as nearest neighbours. Octahedral sites with 4 Fe and 2 Ti atoms are energetically less favourable and become occupied in the γ phase $\text{FeTiH}_{1.9}$ only.

Mg_2NiH_4 goes through a structural phase transformation at 240°C . The high temperature hydride is face centred cubic. The room temperature modification seems to be monoclinic, but up to now the incompleteness of the phase transformation has rendered indexing with a reasonably small unit cell difficult.

The structure of ZrMn_2 hydride can be described by the same space group as the intermetallic ZrMn_2 ($\text{P6}_3/\text{mmc}$). Only interstitials with 2 Zr and 2 Mn atoms are occupied.

Fig. 3 — Crystal structure of FeTiH_x . Hydrogen is located on two different octahedral interstitial sites D1 and D2. The unit cell of the hydride and the smaller unit cell of the intermetallic compound are shown on the left side.



Thermodynamic Properties

The intermetallics which are of interest form hydrides exothermally. In the α phase, the H concentration is proportional to the square root of the H_2 pressure (Sieverts law), small deviations from this being related to H trapping. The plateau pressure, p which varies strongly with temperature, can be described by

$$\ln p = -\Delta S/R + \Delta H/RT$$

where ΔS and ΔH are the changes of entropy and enthalpy, respectively from the α to the β phase. For the limited temperature range considered, ΔS and ΔH can be taken to be temperature independent. Then a plot of $\ln p$ versus T^{-1} yields ΔS and ΔH . The entropy effect is predominantly due to the entropy of H_2 which is lost upon the hydride formation ($-130 \text{ J/K mole H}_2$); ΔS is approximately constant for all hydrides.

Hence for a hydride having a plateau pressure of about 1 bar at room temperature (r.t.) the enthalpy of formation equals $\Delta H = T\Delta S$ and is about -30 to -40 kJ/mole H_2 . For more negative values of ΔH the hydrides are more stable and higher temperatures are required in order to get the plateau pressure of 1 bar.

| | $-\Delta H$ | $T(p = 1 \text{ bar})$ |
|-------------------------------------|-------------------------|------------------------|
| $\text{LaNi}_5 - \text{H}_6$ | 30 kJ/mole H_2 | 15°C |
| $\text{FeTi} - \text{H}_1$ | 27 | 5 |
| $\text{Mg}_2\text{Ni} - \text{H}_4$ | 60 | 280 |
| $\text{ZrMn}_{2+x} - \text{H}_3$ | $50 \div 20$ | $70 \div 170$ |

The plateau pressure at a given temperature can be adjusted over one or two orders of magnitude in off-stoichiometric intermetallics (e.g. ZrMn_{2+x} , $-0.2 \leq x \leq 1.5$) or by substitutions (e.g. $\text{LaNi}_{5-x}\text{Al}_x$, $0 \leq x \leq 1.5$). That is an enormous advantage for the applications.

As was shown by Miedema, the stability of a ternary hydride can be estimated from the stability of the binary hydrides of the components and of the intermetallic. The more stable the intermetallic is, the less stable is the hydride formed. Attempts to estimate the local heat of formation for H in different interstitial sites using this simple Miedema model, have been quite successful:

In ZrV_2 , three types of tetrahedral interstitials can accommodate one H atom. These are sites formed by 2 Zr / 2 V / 1 Zr / 3 V and 0 Zr / 4 V atoms, respectively. In agreement with neutron diffraction data, which show that the 2/2 site is occupied first, the local heat of formation is more negative for the 2/2 sites than for the others. Only for concentrations larger than $\text{ZrV}_2\text{H}_{2.5}$ does the local ΔH for the 1/3 sites become competitive and indeed is the occupation of the 1/3 sites observed.

Diffusion

The diffusibility of interstitial H in many elemental metals is extremely high ($\cong 10^{12}$ jumps/s at r.t.) and exceeds that of heavier interstitials (O,N) by many orders of magnitude. In the simplest jump diffusion model, the diffusion process consists of

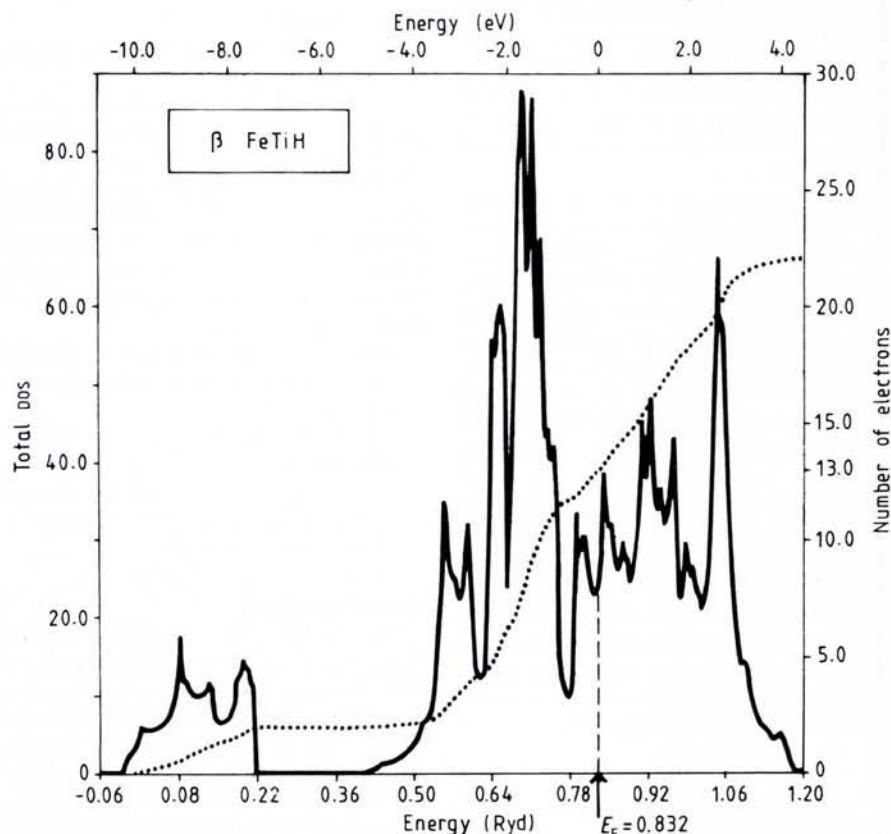


Fig. 4 — Calculated total density of electronic states of FeTiH , (full curve, left scale) and total number of electron (Gupta, *Phys. Lett.* 88A, (1982) 469).

thermally activated steps which are statistically independent. The temperature dependence of the diffusion constant D is given by

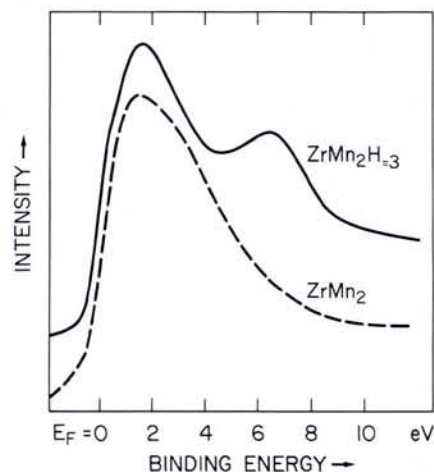
$$D = D_0 \exp(-E_A/kT)$$

where E_A is the activation energy for diffusion.

In intermetallics, the diffusion process is more complicated because of the occurrence of several types of energetically different interstitials. Furthermore, due to the high H concentration, blocking effects become important. Quasielastic neutron scattering and NMR techniques are the widely used experimental methods.

In LaNi_5H_6 two different types of hydrogen motion have been found: A long range diffusion mode ($D = 5 \times 10^{-8} \text{ cm}^2/\text{s}$ at r.t., $E_A = 0.3 \text{ eV}$), and a rapid local motion ($D \approx 2 \times 10^{-6} \text{ cm}^2/\text{s}$ at r.t.) either due to jumps out of and back into the same interstitial, or jumps between two interstitials at a very short distance. The mean residence time is 10^{-4} s . The concentration dependence of the long range diffusion constant is anomalous, i.e. it decreases with decreasing

Fig. 5 — Valence band of ZrMn_2 hydride. The hydrogen absorption induces a new band at 6.5 eV.



H-concentration which is explained by the filling of trap sites at low concentrations.

Although the heats of hydride formation are comparable, the room temperature diffusion in FeTiH is about three orders of magnitude smaller than in LaNi_5H_6 .

The evaluation of the quasielastic neutron scattering data from $\text{Ti}_{1.2}\text{Mn}_{1.8}\text{H}_3$ with a 3 step model (propagation over energetically favourable sites, trapping on less favourable sites, rapid local motion) yields a fast long range diffusion ($D = 10^{-7} \text{ cm}^2/\text{s}$, $E_A = 0.2 \text{ eV}$ at r.t.).

Electronic Structure

Experimental and theoretical work on the electronic properties of d-band metals and hydrides, show that the H absorption results in the modification of the electronic states around the Fermi level, E_F , the formation of a new band about 5 to 9 eV below E_F and a charge transfer between the metal and H. In Pd hydride, e.g., the density of states at E_F is about three times smaller than in Pd, the H induced band appears 7 - 8 eV below E_F and a very small charge transfer from Pd to H occurs, in good agreement with theory and new photoemission results.

Only very recently were the first band-structure calculations and photoemission experiments on hydrides of intermetallics performed.

The method of augmented plane waves (APW) was used to calculate the electronic structure of $\beta\text{-FeTiH}$ (Fig. 4). FeTi is isoelectronic to Cr. This means that E_F falls in the valley of the density of states curve of the split 3d band. E_F of FeTiH is above the valley and accordingly the density of states is much larger than in FeTi . The metal-hydrogen bonding band is centered at 9 eV. It is essentially due to s states of H, but contains also metal d states. Interestingly the Fe d contribution is much larger than that of Ti-d.

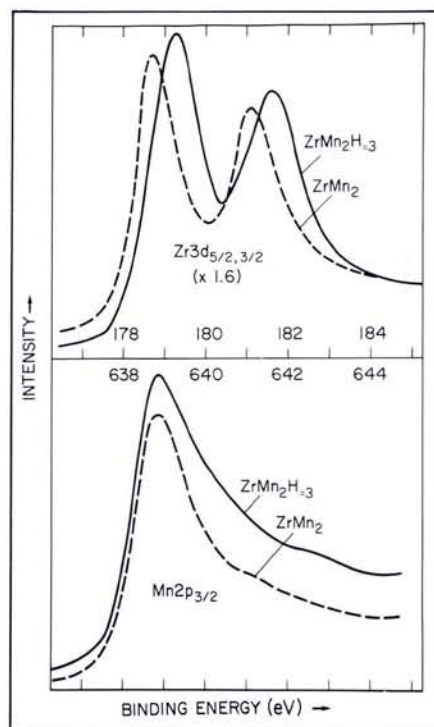


Fig. 6 — Core electron spectra of ZrMn_2 hydride.

The photoemission spectra of ZrMn_2 hydride show (Fig. 5) H induced bonding states at 6.5 eV. The Zr 3d core electrons are chemically shifted to 0.5 eV larger binding energy, in agreement with a strong interaction of Zr with H (charge transfer from Zr to H). There is almost no shift of the Mn 2p core electrons (Fig. 6).

Surface Properties and Activation

The most astonishing difference between the formation of hydrides of elemental metals and of intermetallics is the readiness of intermetallics to absorb H. Whereas a thin oxide layer prevents the H absorption by elemental metals (e.g. Pd), apparently it does not in the case of intermetallics. In the absorption and desorption process, the reaction $\text{Zr}_2 = 2\text{H}$ has to

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proceed at the surface, and it is well known that it proceeds fast on clean surfaces of transition metals.

Surface segregation was found to be a clue to the understanding of the easy H sorption by intermetallics. The component of the lower surface energy (e.g. La in LaNi_5) diffuses to the surface and oxidizes. The remaining d metal (Ni) precipitates so the formation of a compact passivating oxide layer is prevented. H_2 molecules easily penetrate the decomposed surface layer and are thought to dissociate on the metallic subsurface of the intermetallic, particularly on the d precipitates on that subsurface (Fig. 7). Thanks to the ongoing segregation, impurities in the H_2 are chemically bound and a fresh subsurface with fresh d precipitates is formed, even after exposure to air. During the first few absorption/desorption cycles the disintegration of the intermetallic provides further clean surface.

The only hydride forming intermetallic which does not react with H_2 at r.t. is FeTi. It has to be activated at 400°C . It shows no surface segregation at r.t. so that Fe-Ti oxides can be formed at the surfaces. Furthermore the H diffusion is fairly slow. At high temperature and high H_2 pressure, Ti segregates to the surface and forms TiO_2 . The oxidized Fe is chemically reduced to metallic Fe and precipitates together with the Fe which is left by the Ti segregation, in analogy to Ni in LaNi_5 . The surface Fe and Ni precipitates were established by magnetisation measurements, Mössbauer conversion electron spectroscopy and transmission electron microscopy. The Fe precipitates and the FeTi subsurface are highly reactive to dissociate H_2 already at r.t.

Magnetic Properties

H absorption modifies the electronic structure and lattice parameters of the intermetallics and can induce drastic variations of the magnetic moments and of the interaction among them. ZrMn_2 , e.g. appears to be a Pauli paramagnet; the hydride in ferromagnetic with the Curie temperature of 150 K. However, the variation of the bulk magnetism is not the only possible modification of the magnetic properties upon H absorption.

Surface segregation can lead to surface layers of magnetic properties which are quite different from those of the bulk. On compact samples, the amount of surface atoms is so small that their different magnetic behaviour can mostly be neglected. The disintegration of the intermetallics into powder, enlarges the number of atoms in surface layers by several orders of magnitude and their magnetism might dominate the magnetic properties of the bulk. LaNi_5 , e.g. is Pauli paramagnetic with the susceptibility 4.6×10^{-6} emu/g. In the hydride LaNi_5H_6 the susceptibility is 1.3×10^{-6} emu/g. The decrease of the susceptibility is completely covered by the forma-

tion of superparamagnetic Ni precipitations ($T_c = 330^\circ\text{C}$) in a surface layer of $\approx 100 \text{ \AA}$ thickness. In quasistatic susceptibility measurements, the Ni precipitations are superparamagnetic down to 77 K (no hysteresis). Below 77 K they undergo a transition to the normal ferromagnetic state.

Some hydrides of intermetallics tend to disproportionate into a stable binary hydride and another intermetallic of different magnetic properties. Th_7Fe_3 , which is superconducting below 2K, was reported to form a ferromagnetic hydride. But recently a more careful analysis revealed that the observed ferromagnetism was not due to the formation of the ternary hydride, but due to the disproportionation into Th hydride and a Fe richer ferromagnetic Th-Fe compound.

Thus, the study of the magnetic properties of hydrides of intermetallics is exciting, but a careful analysis of the samples before and after the H absorption and desorption and also of the reproducibility of the results is strongly recommended to separate reversible from irreversible effects.

Kinetics

H absorption occurs over several steps. The H_2 flows through cracks to the surface where the H_2 molecules have to dissociate. H atoms enter the bulk and diffuse through the metal lattice to find a nucleation centre where the α - β phase transition proceeds. Furthermore the heat of reaction has to be dissipated. Which of the three processes: gas flow, intrinsic kinetics ($\text{H}_2 \rightleftharpoons 2\text{H}$, surface penetration, diffusion, phase transformation) or heat flow is the slowest and controls the overall kinetics?

For most hydrogen storage systems on a technologically interesting scale, in which activated intermetallics are used, heat transfer is rate controlling. The thermal contact between the powdered intermetallic and the reactor bed is insufficient so that the heat of reaction heats up the material. Though the higher temperature accelerates the intrinsic kinetics, it also raises the plateau pressure until it equals the gas pressure of the system and the reaction slows down. The heat transfer could be improved successfully by adding Al or Cu to the powder of the intermetallic and by pressing the mixture to pellets which fit heat exchanger tubes. The limited heat transfer, on the other hand, provides safety: If the storage bed leaks, the starting desorption cools the intermetallic and slows down the intrinsic kinetics.

On nonactivated samples, the reaction $\text{H}_2 \rightleftharpoons 2\text{H}$ is blocked at the surface. This is often the case for binary hydrides if the samples are coated with a passivating oxide layer. Most intermetallics are self-activated by surface segregation and by the formation of a fresh surface upon fragmentation. But the surface reaction can become rate controlling again if the

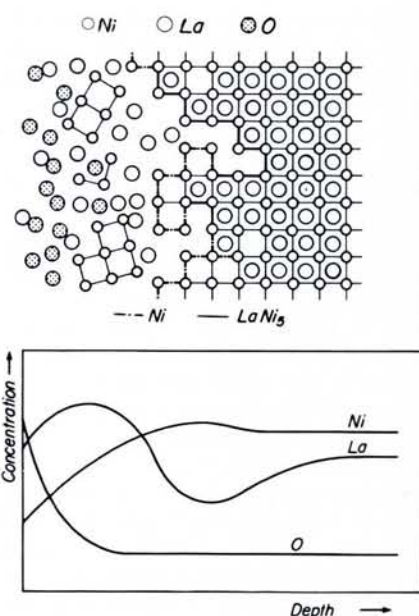


Fig. 7 — Segregated surface layer of LaNi_5 and corresponding depth profile. The Ni precipitates at the subsurface and the subsurface itself are able to dissociate H_2 .

hydrogen gas contains significant amounts of impurities such as SO_2 , H_2S and CO.

The diffusion would be rate determining in large bulk samples of FeTi, e.g. In the widely used FeTi powder of grain size $\approx 1 \mu\text{m}$, the diffusion path is short so that the diffusion is fast enough. High purity MgH_2 and also Mg_2NiH_4 are among the few materials in which the room temperature diffusion is far too low. But already their thermodynamic properties require high temperature.

Mass flow becomes dominant in materials which easily sinter together such as CaNi_5 or Mg compounds at high temperature.

Applications

The application of hydrides of intermetallics is not limited to the well-known mobile or stationary storage of H_2 as a carrier of secondary energy (automobiles, peak shaving). Many other applications, often related to energy technology, are feasible:

- chemical heat pump or thermochemical compressor;
- selective absorption of H_2 from gas mixtures; H_2 purification; supply of ultra-pure H_2 at constant pressure;
- electrochemical cells;
- catalysts for hydrogenation reactions (synthetic fuels);
- isotope separation.

As these applications have been described recently in several papers (e.g. *Scientific American* **242** (1980), p. 118) we get to the question, whether degradation effects are known which limit the cyclic lifetime of the intermetallics.

In open systems, in which fresh H_2 is admitted with each cycle, H_2 impurities may not only slow down the kinetics, but induce the surface decomposition of the in-

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termetallics and thus gradually decrease the storage capacity. A pure LaNi_5 storage unit, e.g., when cycled in $\text{H}_2 + 0.5\% \text{O}_2$ at r.t., loses about 30% of its capacity within 100 absorption/desorption cycles due to the decomposition of LaNi_5 into La_2O_3 and Ni. The binding of the impurities to the intermetallic is of course a purification of H_2 . The addition of some Al improves the stability of LaNi_5 towards surface decomposition considerably: $\text{LaNi}_{4.7}\text{Al}_{0.3}$ is less reactive with oxygen and the losses of capacity at room temperature about 10 times smaller. The surface decomposition is of course harmless in closed systems, in which the same H_2 is absorbed and desorbed cyclically. However, when H-absorption/desorption was controlled thermally at constant pressure, the (reversible) disproportionation of LaNi_5 into La hydride and Ni was observed above 200°C .

FeTi has been run in a closed system over 30000 cycles without noticeable deterioration. In Mg_2Ni , sintering at the rather high temperature of desorption can cause problems.

REFERENCE

For references the reader is referred to the "Proceedings of the Int. Symp. on Properties and Applications of Metal Hydrides", *J. Less Common Metals* **73**, **74** (1980) and **88**, **89** (1982).

The annual meeting of the European Group for Atomic Spectroscopy (EGAS), organized by Professor N. Grevesse and coworkers, took place in the last week of July 1982, at the University of Liège. We were more than 150 participants from 16 countries. The traditional style of EGAS meetings of having a small number of invited talks (five in this case) and many contributed papers and poster sessions which were included for the first time at Orsay in 1979 was followed. Poster sessions have become popular because they give more time for discussion and they avoid parallel sessions which always pose problems to those who wish to concentrate on the topics that concern them personally.

Laser spectroscopy continues to play a dominant role at EGAS meetings. H. Walther reported on recent advances in this area: heterodyne detection of Rydberg atoms' maser emission, single atom detection, hfs and transfer collisions between states, etc.

The problem of hertzian coherence in spectroscopy can be considered a normal topic for EGAS meetings, and now the interest in nonlinear aspects is increasing. W. Lange discussed different recent techniques: sideband spectroscopy, pulse train spectroscopy, quantum beats in transmission. Other results, such as the pressure-induced Hanle-effect, were also covered.

Particle accelerators with fast ion beams have become in the past decade a clearly identified field of atomic physics. In particular, the beam foil spectroscopy technique is one of the most versatile and powerful methods of measuring excitation and ionization energies. J. Désesquelles reviewed developments in this area with emphasis on highly ionized atoms and highly excited states: Rydberg and multiple excited levels; f-value measurements in iso-electronic systems with a small number of valence electrons and in elements of solar or stellar importance, and zero field quantum beat fine structure measurements.

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