In order to improve the compressor cycling stability and hydrogen storage properties of V_{40}Ti_{21.5}Cr_{33.5} alloy further, the fourth element i.e. Niobium, Iron, or Zirconium has been substituted for Cr to prepare V_{40}Ti_{21.5}Cr_{33.5}M_{5} alloy and their cyclic hydrogen absorption-desorption performance was evaluated up to 100 cycles for temperature and pressure ranges of 20-300 °C and 5-20 MPa, respectively. All the three compositions have shown different sorption properties as well as cyclic stability. The V_{40}Ti_{21.5}Cr_{33.5}Nb_{5} alloy was found a most suitable composition with a comparatively high hydrogen capacity and reasonable stability after 50 cycles of compressor cyclic test, whereas, V_{40}Ti_{21.5}Cr_{33.5}Fe_{5} alloy has shown worst cyclic stability with lowest hydrogen capacity. However, structural and morphological investigations suggest no phase segregation during the cycling of V_{40}Ti_{21.5}Cr_{33.5}Fe_{5} alloy in contrast to the other alloys, which suggest that phase segregation is not the only responsible reason for the performance degradation of BCC alloys employed for compressor cycle. In some cases similar to V_{40}Ti_{21.5}Cr_{33.5}Fe_{5}, stress/strain formation in the lattice during cycling may also cause the degradation of the material.

1. Introduction
Thermochemical hydrogen compressor using metal hydride system offers several operational and economic advantages over the traditional mechanical hydrogen compression. A typical metal hydride based hydrogen compressor uses a metal which absorbs low-pressure hydrogen gas at an ambient temperature and releases high-pressure hydrogen gas at higher temperatures. Vanadium based BCC alloys are considered as promising candidates for the development of metal hydride based...
hydrogen compressor which has high hydrogen storage capacity (~ 4 wt %). In connection with this, several compositions of V with different metals particularly Ti and Cr, have been extensively studied for their better hydrogen storage properties. The cyclic durability of hydrogen absorption and desorption of metal hydride is very important for practical application of metal hydride towards hydrogen compressor. However, the gradual reduction in hydrogen capacity of these materials over few cycles have not been understood yet and so could not be resolved to the desired level. The alloying elements and their compositions are known to tune these properties efficiently. Our recent studies of hydrogen storage properties and cyclic compressor performance of V<sub>t</sub>Cr<sub>x</sub> Ti<sub>x</sub> and V<sub>t</sub>Cr<sub>y</sub> Ti<sub>y</sub> alloys up to 100 cycles suggested that the alloy with less V content undergoes through degradation continuously over number of cycles, while higher V containing alloy becomes stable after 10 cycles. A phase segregation of relatively stable hydrides i.e. V<sub>H2.5</sub> and Ti<sub>H0.6</sub> were proposed responsible for this capacity degradation of less vanadium alloy. In general, the addition of the fourth element to V-Ti-Cr alloy has been shown to be an effective way to improve the cyclic durability as well as hydrogenation properties of these alloys. Towata et al. studied the cyclic durability of the Ti<sub>30</sub>Cr<sub>20</sub>V<sub>50</sub> alloy at room temperature and found 5% Nb substitution effective to improve hydrogen storage capacity and cycle durability. Aoki et al. studied that the Fe substitution also improved the cyclic durability of Ti-Cr-V system but the effective hydrogen storage capacity was decreased during cycling. Yan et al. found the highest effective hydrogen capacity 3.78 wt% for the V<sub>t</sub>Cr<sub>x</sub>Ti<sub>y</sub>Fe<sub>0.5</sub> alloy with a reversible capacity of 2.26 wt%. Zr substitution has also been found effective to improve the cyclic performance of Ti-Cr-V alloy and the maximum capacity was found as 3.19 wt% at 2.0 MPa and 298 K for Ti<sub>0.43</sub>Zr<sub>0.07</sub>Cr<sub>0.25</sub>V<sub>0.25</sub> alloy. Motivated from the performance of Niobium (Nb), Iron (Fe), and Zirconium (Zr) as the substituting elements, V<sub>t</sub>Cr<sub>x</sub> Ti<sub>y</sub> alloy has been modified by the substitution of Cr by the above elements in order to further improve its cyclic stability and sorption capacity. Since it is necessary to study the hydrogen sorption properties of these alloys at high temperature and high pressure to employ it for metal hydride hydrogen compressor, this work focuses on the effect of Nb, Fe, and Zr substitution on the cyclic compressor performance of V<sub>t</sub>Cr<sub>x</sub> Ti<sub>y</sub> up to 100 cycles as well as their sorption properties before and after compressor test in terms of structural and morphological properties.

2. Experimental

Nb, Fe and Zr substituted alloys V<sub>t</sub>Cr<sub>x</sub> Ti<sub>y</sub>Nb<sub>z</sub>, V<sub>t</sub>Cr<sub>x</sub> Ti<sub>y</sub>Fe<sub>z</sub> and V<sub>t</sub>Cr<sub>x</sub> Ti<sub>y</sub>Zr<sub>z</sub> were purchased from Japan Metals and Chemicals Co. Ltd. As per our need, the materials were prepared by arc melting of the starting elements in the respective molar ratio with 99.9% purity. To ensure homogeneity the samples were melted three times under argon atmosphere. The samples were annealed at 1500 K for 24 h to improve the crystallinity and to dissolve some minor phases. The SR-XRD measurements were performed by using a large Debye-Scherer camera with an imaging plate at the beamline BL5S2 General Material Evaluation I (X-ray diffraction) (Aichi Synchrotron Radiation Center). The samples used for the SR-XRD experiments were stuffed in glass capillaries with 0.01 mm in thickness, 0.50 mm in diameter and 80 mm in length (Hilgenberg), which were sealed with an epoxy adhesive. The wavelength of the X-ray beam used in this study was 1.0 Å calibrated by the lattice constant of ceria CeO₂ at room temperature and the energy output was 12.4 keV. The SR-XRD data has been converted using Bragg's equation and has been shown corresponding to the CuKα wavelength (1.5406 Å) for a direct comparison to powder XRD data. Scanning Electron Microscopy (SEM) on JEOL, JSM-6380A instrument was performed to observe the powder morphology of the samples. The pressure-composition isotherms (PCI curves) were measured by Sieverts-type apparatus (Suzuki Shokan Co. Ltd.) in the temperature and pressure range of 22 °C (denoted as RT hereafter) -100 °C and 0001-5 MPa, respectively. An initial activation process was performed before making PCI measurements and compressor cycle test. For the activation, the alloy powder was exposed to 5 MPa hydrogen at room temperature (RT) which was increased up to 100 °C and kept it at this temperature for 4 h, then the sample holder was evacuated and kept for 2 h under dynamic vacuum. This whole process was repeated 3 times to activate the sample completely. Finally, the sample was kept at 200 °C for 6 h under dynamic vacuum to ensure complete hydrogen desorption. The compressor cyclic tests were performed in a closed sample holder by achieving 20 MPa in temperature range RT-300 °C. The initial pressure at room temperature (22 °C) for compressor tests was kept at 2 MPa for all the alloys similar to their parent alloy V<sub>t</sub>Cr<sub>x</sub> Ti<sub>y</sub>. The procedure to perform compressor cyclic test has been described elsewhere.
3. Results and discussion

Fig. 1 shows the SR-XRD profiles for all the substituted V_{40}Ti_{21.5}Cr_{33.5} alloys, indicating the existence of only BCC structure same as that of their parent element vanadium for all the prepared alloys except Zr substituted alloy. In addition to the main BCC phase, as prepared V_{40}Ti_{21.5}Cr_{33.5}Zr_{5} alloy shows the additional phase of ZrCr_{2} which is crystallized in cubic structure as shown in Fig. 1 (b). The peaks corresponding to the main V based BCC phase in SR-XRD profiles are found to be shifted to lower angle (shown by down arrow) after 100 cycles of compressor test for all the alloys, which is a sign of lattice expansion due to residual hydrogen left in the samples, even if the alloys were heated at 100 °C under vacuum for 2 h. In addition, the peaks are broadened after cycling which must be due to crystallite refinement during compressor cycles. The behavior of substituted alloys has been found quite similar to the parent alloy in terms of peak shift due to residual hydrogen \[ \text{\textsuperscript{20}} \]. In addition to the peak shift in SR-XRD profile, Nb substituted sample i.e. V_{40}Ti_{21.5}Cr_{33.5}Nb_{5} shows the peak shape change especially at around 40° for SR-XRD which could be indexed by BCT - VH_{0.81} phase as shown in Fig. 1 (a) in addition to BCC phase with residual hydrogen (shown by down arrow). The existence of the peaks corresponding to both phases, very near to each other forms the assymetric shape of this peak at around 40°. It is important to note here that VH_{0.81} is representing the BCT phase only according to XRD database, irrespective to its exact hydrogen content. The actual hydrogen content may be different in range of 0.5-1.0. Apart from these, the SR-XRD (Fig. 1 (b)) profiles of Zr substituted sample i.e. V_{40}Ti_{21.5}Cr_{33.5}Zr_{5} alloy suggests the existence of additional peaks at around 35°, 40° which could be indexed by the ZrV_{2} phase. Thus the laves phase of ZrCr_{2} in the starting sample transformed to ZrV_{2} phase during the hydrogen sorption compressor cyclic test as shown in Fig 1 (b). In addition to these phases, the original BCC phase could also be observed with a slight shift to lower angle, which is associated with the residual hydrogen left in the sample. The morphology of all substituted V-Ti-Cr alloys is shown in Fig. 2, which suggests the reduction in particle size during compressor cycling for all alloys. Niobium substituted sample shows the least pulverization during cycling, where

![Fig. 1 SR-XRD profile for Pristine and cycled (a) V_{40}Ti_{21.5}Cr_{33.5}Nb_{5}; (b) V_{40}Ti_{21.5}Cr_{33.5}Zr_{5}; (c) V_{40}Ti_{21.5}Cr_{33.5}Fe_{5} alloys. Here all the alloys are V-based solid solution having BCC structure of their parent vanadium metal; V, VH_{5} (vanadium structure having residual hydrogen), VH_{0.81} (BCT structure) are used only from the structure point of view](image)

![Fig. 2 SEM morphology for Pristine (left) and cycled (right) (a) and (b) V_{40}Ti_{21.5}Cr_{33.5}Nb_{5}; (c) and (d) V_{40}Ti_{21.5}Cr_{33.5}Zr_{5}; (e) and (f) V_{40}Ti_{21.5}Cr_{33.5}Fe_{5} alloys](image)
the highest pulverization is observed for Zr-substituted sample.

Fig. 3 (a), (b), and (c) show the compressor cyclic test for V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅, V₄₀Ti₂₁.₅Cr₃₃.₅Zr₅, and V₄₀Ti₂₁.₅Cr₃₃.₅Fe₅ alloys, respectively. The required temperature to achieve the target pressure of 20 MPa is found to be 217 °C for Nb substituted V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅ alloy, 255 °C for V₄₀Ti₂₁.₅Cr₃₃.₅Zr₅ alloy and 290 °C for Fe substituted V₄₀Ti₂₁.₅Cr₃₃.₅Fe₅ alloy. The compressor cyclic test data for only first 10 cycles is shown here to make it clear to the readers. Fig. 3 (d) shows the cyclic stability of the alloys up to 100 cycles as a function of H₂ pressure in the sample holder at room temperature. The pressure increase inside the sample holder at RT is a direct measure of remained excess gaseous hydrogen, which could not be absorbed by the material in each successive cycle. This can be used to estimate the degradation of the material. The pressure in the sample holder containing Nb substituted V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅ alloy is found to be increased from 2 MPa to 4.85 MPa continuously in first 50 cycles and then become stable up to 100 cycles. The degradation can be explained on the basis of phase separation during the cyclic test. The SR-XRD profiles (Fig. 1 (a)) suggest the existence of V₁H₀.₈₈ phase which is formed gradually during cycling. This phase cannot desorb hydrogen at studied temperature range and remain in the alloy as dead mass for the remaining cycles, which causes the capacity decay in successive cycles. Once this phase is completely segregated, the capacity becomes stable which causes a stable pressure after 50 cycles. The pressure in the sample holder containing Zr substituted V₄₀Ti₂₁.₅Cr₃₃.₅Zr₅ alloy is found to be increased from 2 MPa to 4 MPa gradually in the first 25 cycles and then became almost stable up to 100 cycles. The capacity decay in the first 25 cycles must be due to the transformation of ZrCr₂ laves phase to ZrV₂ phase through a tie line of ZrCr₂ - Zr(Cr₁₋ₓVₓ)₂ - ZrV₂ gradually as evidenced from SR-XRD profiles shown in Fig. 1 (b). Since cubic ZrCr₂ phase can absorb a high amount of hydrogen (4 H/f.u.) ²⁶, thus contribute towards the overall initial hydrogen capacity of the material, however, it forms very stable hydride and cannot desorb hydrogen easily. Moreover, gradually formed ZrV₂ phase forms even more stable hydride ²⁷, thus the hydride of all these materials i.e. ZrCr₂ - Zr(Cr₁₋ₓVₓ)₂ - ZrV₂ act as dead mass and lower the overall capacity of the material. The Fe substituted sample has shown the worst performance towards cyclic stability in contrast to the earlier reports of nice stability at room temperature. The pressure in the sample holder is found to be increased continuously up to 100 cycles and reach to 7.5 MPa from 2 MPa. This shows that the Fe substituted alloy undergoes a continuous degradation during cycling.

Fig. 3 Cyclic compressor tests of (a) V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅, (b) V₄₀Ti₂₁.₅Cr₃₃.₅Zr₅, (c) V₄₀Ti₂₁.₅Cr₃₃.₅Fe₅ alloys, (d) performance comparison of all alloys in terms of system pressure variation at room temperature with no. of cycles.
Surprisingly, no phase segregation could be observed from SR-XRD (Fig. 1 (c)). This behavior is in contrast to that for V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅ and V₄₀Ti₂₁.₅Cr₃₃.₅Fe₅ alloys, where the degradation was caused by the phase segregation and formation of stable Ti and V hydride phases. The possibility of this degradation can be associated with a different mechanism based on the stress/strain or dislocation defects development in the lattice during the compressor cycling as suggested earlier. Kim et al. have shown an increase in the number of dislocation defects in BCC hydrides using the pair distribution function (PDF) analysis. It is suggested by them that the capacity degradation can be reduced by designing the alloys forming bct structured hydrides. This can be observed very clearly in our experiments on Nb and Fe substituted alloys. A BCT monohydride phase (\( \sim \) VH₀.₈₁) is formed after cycling in the Nb substituted sample, which must have reduced the formation of lattice defects during cycling, thus became stable after initial decay of capacity due to stable VH₀.₈₁ formation as discussed above. On the other side, Fe substituted alloy doesn’t form BCT monohydride phase as evidenced from SR-XRD results (Fig. 1 (c)). It forms only BCC hydride phase which is quite easy to produce lattice defects in each successive cycle, thus continuing the capacity degradation.

The continued degradation of V₄₀Ti₂₁.₅Cr₃₃.₅Fe₅ and the initial decay of V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅ and V₄₀Ti₂₁.₅Cr₃₃.₅Zr₅ alloys can also be confirmed from the PCI experiments performed after 10, 25, 50, and 100 cycles of compressor test. Fig. 4 shows PCI curves at RT for V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅, V₄₀Ti₂₁.₅Cr₃₃.₅Fe₅, and V₄₀Ti₂₁.₅Cr₃₃.₅Zr₅ alloys. It is clear from Fig. 4 (a) that the plateau region is shifted to lower pressure for Nb and Zr substituted alloy in comparison to its parent alloy, whereas an opposite shifting to higher pressure is observed for Fe substituted sample. The Nb substituted V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅ alloy has clear absorption and desorption flat plateau regions as compared to the other two alloys. The plateau pressure value at the middle point of plateau region are 0.28 MPa and 0.028 MPa for absorption and desorption, respectively, which are lower than that of its parent alloy V₄₀Ti₂₁.₅Cr₃₃.₅. This is associated with the lattice expansion due to higher atomic radii of Nb (198 pm) in comparison to Cr (166 pm), thus allowing easy hydrogen accommodation in the lattice resulting in lowering the plateau pressure. In addition, the hydrogen storage capacity of the V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅ alloy is found to be 2.5 wt% in the first cycle which is slightly higher than the parent alloy (2.35 wt% [20]). The capacity is...
found to be gradually decreased down to 2.08 wt% during 100 compressor cycle test (Fig. 4 (b)). The same trend has been observed for Zr substituted alloy due to its larger atomic radii (206 pm) with absorption and desorption plateau at 0.209 MPa and 0.016 MPa, however, the slope is slightly larger than that of Nb substituted alloy. In addition, the decay in the maximum capacity of the V₄₀Ti₂₁.₅Cr₃₃.₅Zr₅ alloy is found to be from 2.3 wt% to 1.93 wt% during first 25 cycles (Fig. 4 (c)). After 25 cycles the PCI curves are found to be quite similar up to 100 cycles which confirms the cyclic stability of this alloy. Moreover, a strange zigzag shape has been observed in the absorption PCI curves of Nb and Zr substituted alloy. This should be due to tight packing of a large amount of sample (13 g) in a small volume, thus hindering the sample surface partially from the quick exposure with hydrogen. During the stabilizing time of pressure, some of the hydrogen could be absorbed slowly and thus reduce the pressure. It generates a pressure difference between two successive judging time when the instrument starts to record the pressure value and gives rise to zigzag shape in PCI. This zigzag shape has been disappeared, when the PCI measurements were performed.

![Fig. 5 PCI curves at different temperatures for V-Ti-Cr alloys; (a) pristine & (b) Cycled V₄₀Ti₂₁.₅Cr₃₃.₅Nb₅; (c) pristine & (d) Cycled V₄₀Ti₂₁.₅Cr₃₃.₅Zr₅; (e) pristine & (f) Cycled V₄₀Ti₂₁.₅Cr₃₃.₅Fe₅. Closed symbols: Absorption, Open symbols: Desorption](image)
with 1 g sample in the same volume sample holder (Fig. 4). On the other hand, Fe substituted V_{40}Ti_{21.5}Cr_{33.5}Fe_{5} alloy has higher plateau pressures at 3.808 MPa and 0.685 MPa for absorption and desorption, respectively (Fig. 4 (a)). This is due to the contraction of lattice resulting from the smaller atomic radii of Fe (156 pm), thus making it difficult for hydrogen atoms to accommodate in the lattice and increase the plateau pressure in comparison to its parent alloy. The maximum H\textsubscript{2} storage capacity of the V_{40}Ti_{21.5}Cr_{33.5}Fe_{5} alloy is found to be 1.878 wt\%, which is less than that of its parent alloy, which further decreased to 1.00 wt\% after 100 cycles (Fig. 4 (d)). In addition to this, PCI curves are found to be quite similar after 50 and 100 cycles.

Fig. 5 shows the PCI curves for all the alloys (1 g sample) at RT, 50 °C, and 75 °C before and after compressor cycling. The absorption and desorption plateau pressure at RT is found to be 0.21 MPa and 0.03 MPa for the pristine V_{40}Ti_{21.5}Cr_{33.5}Nb_{5} alloy which is slightly reduced to 0.07 MPa and 0.025 MPa for cycled V_{40}Ti_{21.5}Cr_{33.5}Nb_{5} alloy, with a respective maximum capacity 2.63 wt\% and 2.02 wt\% as shown in Figs. 5 (a) and (b). The Zr substituted pristine V_{40}Ti_{21.5}Cr_{33.5}Zr_{5} alloy shows the absorption and desorption plateau pressure at 0.08 MPa and 0.01 MPa with a maximum capacity of 2.5 wt\% with the reversible capacity of 2 wt\% (Fig. 5 (c)). The plateau slope has been increased after compressor cycling, however still maintaining its maximum capacity as 1.75 wt\% at RT as shown in Fig. 5 (d).

In case of Fe substituted sample, PCI shows the maximum capacity 2.46 wt\% with the absorption and desorption plateau pressure at 3.68 MPa and 0.86 MPa as shown in Fig. 5 (e). Moreover, no plateau region could be observed at higher temperatures i.e. 50 and 75 °C because it must be at much higher pressure than the studied range. The plateau region at RT has been shortened drastically after 100 cycles of compressor test as visible from Fig. 5 (f).

Fig. 6 shows the van’t Hoff plot for the parent and Nb, Zr substituted alloys pristine samples which are used to obtain the thermodynamic parameters of enthalpy (\Delta H) and entropy (\Delta S) changes. The enthalpy and entropy values for Fe substituted V_{40}Ti_{21.5}Cr_{33.5}Fe_{5} alloy could not be calculated as no plateau region could be appeared at high temperatures (Fig. 5 (e)). The enthalpy and entropy values are found to be -47 kJ mol\(^{-1}\) and 147 J mol\(^{-1}\)K\(^{-1}\) H\textsubscript{2} for substituted V_{40}Ti_{21.5}Cr_{33.5}Nb_{5} alloy and -53 kJ mol\(^{-1}\) and 159 J mol\(^{-1}\)K\(^{-1}\) H\textsubscript{2} for substituted V_{40}Ti_{21.5}Cr_{33.5}Zr_{5} alloy in comparison to the enthalpy and entropy values -41 kJ mol\(^{-1}\) and 146 J mol\(^{-1}\)K\(^{-1}\) H\textsubscript{2} of parent V_{40}Ti_{21.5}Cr_{38.5} alloy.\textsuperscript{20} The higher enthalpy value indicates the lower dissociation pressure, higher stability of the hydride and higher dehydrogenation temperature. The higher entropy value indicates a higher disorder of H\textsubscript{2} in the host lattice.

4. Conclusions

Hydrogen sorption and cyclic compressor performance of V_{40}Ti_{21.5}Cr_{33.5}M_{5} (M= Nb, Zr, Fe) alloys has been systematically studied. The V_{40}Ti_{21.5}Cr_{33.5}Nb_{5} and V_{40}Ti_{21.5}Cr_{33.5}Zr_{5} alloys were found stable after 50 and 25 cycles of compressor test. The initial degradation was suggested to be associated with the formation of stable hydride phases i.e. VH_{0.81} and ZrV_{2}H_{x}. On the other hand, V_{40}Ti_{21.5}Cr_{33.5}Fe_{5} alloy was found to be degraded continuously up to 100 cycles and hydrogen storage capacity was found to be decreased after each cycling. It is in contrast to the earlier reports at room temperature which suggest enhanced stability at room temperature with Fe substitution. However, the phase segregation could not be observed for Fe substituted alloy, in contrast to Nb and Zr substituted alloys, suggesting that the disproportionation is not always the reason for capacity fading of these BCC alloys. The stress/strain generation in the lattice due to elevated temperature and pressure conditions during cycling seems more appropriate for this degradation. Finally, it is concluded that Nb substituted alloy among all these V-Ti-Cr alloys has the best hydrogenation
properties and stability towards high temperature and pressure conditions in terms of its absorption and desorption capacity and cyclic durability.

Acknowledgment

This work was supported by New Energy and Industrial Technology Development Organization (NEDO), New Energy Venture Business Technology Innovation Program (Fuel cell・Storage cell), “Development of energy-saving hydrogen compressor by renewable energy”. The authors would like to acknowledge Dr. Shin-ichi Towata in Aichi Synchrotron Radiation Center for performing Synchrotron X-ray Diffraction.

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