Mechanical fracture of vanadium dioxide during thermal cycling

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Abstract

The degree of vanadium dioxide VO\textsubscript{2} mechanical fracture and solid solutions on its base V\textsubscript{1-x}Fe\textsubscript{x}O\textsubscript{2} is quantified. Dependences of graded powder mass fraction m/M on the number of thermal cycles, affected by VO\textsubscript{2} sample, and for vanadium dioxide samples containing small quantity of iron (solid solutions V\textsubscript{1-x}Fe\textsubscript{x}O\textsubscript{2}) are obtained. Dependence of fracture degree and fractional change of cell volume on the sample V\textsubscript{1-x}Fe\textsubscript{x}O\textsubscript{2} composition for samples, tested by 50 thermal cycles, is given. It is found out that the fracture degree is the lowest for the sample, containing 1\% of iron.

Keywords: mechanical fracture; thermal cycling; phase transition; vanadium dioxide

1. Introduction

Vanadium dioxide and solid solutions on its base, as well as other compounds of 3d-, 4d- and 5f- elements, under temperature stress experience phase transitions “metal-semiconductor” (PTMS). Material is a semi-conductor below transition temperature, when the temperature is higher, it obtains metal properties. Under transition, various sample properties (compositional, electrical, magnetic, thermophysical, optical and others) change, which is widely applied in engineering.

Thanks to rather easy technology of their preparation (manufacturing) and practically convenient transition temperatures T\textsubscript{ms} (~340 K and higher), vanadium dioxide-based compounds are used more often than others. These materials are used for producing in-rush current limiters in electro-technical and electrical devices, as well as

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thermo-resistors, thermorelays, and automatic control devices. Composition and properties of solid solutions and vanadium dioxide-based compounds depend on various factors, both technological and physic-chemical. The study of VO\textsubscript{2} properties provides its usage as a detector for hydrocarbon accumulations exploration and the following development of new fields, as well as for the estimation of oil wells condition.

2. Study subject

There is a big problem concerning the practical usage of vanadium dioxide and solid solutions on its base because of its mechanical degradation, resulting from structuring transformation during PTMS. It is known, that VO\textsubscript{2} cell volume changes considerably during phase transition [1, 2], and this results in high internal stress and can lead to mechanical fracture of the sample.

3. Methods

Simple and direct method of quantitative measurement of material mechanical fracture degree is a calibrated sieve analysis. Our aim is not to define the size range of the particles, composing powder sample of vanadium dioxide because there is no need to do it. We have to find out if there is noticeable, recorded material fracture or not. It is very easy to do, if to take the part of the powder which has not been sieved at first, subject it to thermal cycling and sieve it again. If there is a newly-sieved powder, the material fracture is observed.

The sieve with mesh size 0.1 \times 0.1 \text{mm}^2 was certified in accordance with ISO 3310. The registration was made by the following sequence: initial quantity with mass \(M\) was sieved. The mass left in the screen was weighed and, by simple subtraction, mass of graded powder \(m\) was found. Then, not-graded mass was subjected to thermal cycling, was sieved and then the mass of graded powder was found again.

4. Results and discussion

The mass of graded powder “m” was found out increasingly after every thermal cycling. To gather statistical data, the experiment was repeated three times under different initial sample masses. Fig. 1 presents the dependence of the graded powder VO\textsubscript{2} fractional mass on the number of thermal cycles, affected by the sample. As it can be seen from the obtained results, it is true that thermal cycling results in mechanical fracture of vanadium dioxide. The more the number of thermal cycles affected by a sample, the bigger the fracture. Non-linearity of the dependence \(<m>/ <M>\) on the number of thermal cycles, in our view, may result from the fact that the particles of the coarse fractions are divided into particles, the size of which does not allow them to "pass" through a sieve, and the number of particles forming the sieved particles decreases with each cycle.
Fig. 1. Mass fraction of graded powder VO₂ m/M depending on the number of thermal cycles affected by the sample

Fig. 2 shows the research results of the thermal cycling influence on the vanadium dioxide samples, containing small quantity of iron (solid solutions V₁₋ₓFeₓO₂).

Fig. 2. Mass fraction (γ = m/M) of graded powder V₁₋ₓFeₓO₂ depending on the number of thermal cycles affected by the sample
As it can be seen from the obtained results, for samples $V_{1-x}Fe_xO_2$, irrespective of their iron content, mechanical disruption during thermal cycling is also observed. However, it should also be noted, that, the more the iron content in the sample, the higher the degree of its mechanical disruption (under the given number of thermal cycles). The conclusion was made on the basis of $<\gamma>$ change with the alteration of the sample composition.

Fig. 3 shows the dependence of the disruption degree $<\gamma>$ and fractional cell volume change $\Delta V/V$ on the sample $V_{1-x}Fe_xO_2$ ($N = 50$) composition for the samples, tested by 50 thermal cycles. Changing in cell volume was observed during X-ray diffraction analysis. More detailed results of this research are submitted for a publication.

As it can be seen from Fig. 1, $<\gamma>$ falls from 0.78 for pure vanadium dioxide to 0.61 for $V_{0.99}Fe_{0.01}O_2$, and then grows up to 0.74 for $V_{0.93}Fe_{0.07}O_2$. This fracture degree dependence $<\gamma>$ correlates well with the dependence of fractional cell volume change $\Delta V/V$ on the sample $V_{1-x}Fe_xO_2$ composition.

5. Conclusion

In conclusion, the fracture degree both for pure vanadium dioxide and for iron-containing sample $V_{0.93}Fe_{0.07}O_2$ is rather high; for sample $V_{0.99}Fe_{0.01}O_2$ it is lowest. Thus, it is more efficient to use the sample with the lowest fracture degree in practice.

References