J Therm Anal Calorim (2010) 101:447–453
DOI 10.1007/s10973-010-0781-1

The reactivity of $SbVO_5$ with T-Nb_2O_5 in solid state in air

Elzbieta Filipek · Mateusz Piz

CCTA10 Special Issue

Abstract The reactivity of $SbVO_5$, a compound known since a short time, with T-Nb_2O_5 in solid state in air has been investigated over the whole component concentration range of a system built by these two reacting substances. The investigation results have shown that an equimolar mixture of $SbVO_5$ and T-Nb_2O_5 reacts with a subsequent formation of a hitherto unknown compound of the formula Nb_2VSbO_{10}. This compound has been characterized by the methods XRD, DTA/TG, and SEM. Its orthorhombic unit cell parameters have been calculated, and its stability in air up to 880 ± 10 °C has been proved. At this temperature, the compound melts incongruently with an accompanying deposition of solid Nb_9VO_{25}, i.e., of a compound that crystallizes in the binary oxide system V_2O_5–Nb_2O_5.

Keywords Niobium–vanadium–antimony-oxides · Solid–solid reaction · Nb_2VSbO_{10} · XRD · DTA · SEM/WDX

Introduction

Oxides of the IIIb and Vb family elements of the periodic table as well as compounds formed in binary and ternary systems built by these oxides have been already for several decades an attractive object of various investigations which have proved that these oxides and compounds evince properties interesting for possible applications, including catalytic, optical, electrical, and magnetic properties.

It is known, among other things, that both vanadium(V) oxide and niobium(V) oxide as well as compounds and phases formed through reactions between these oxides (i.e., $NbVO_5$, Nb_9VO_{25}, $Nb_{18}V_4O_{55}$, and the solid solution of V_2O_5 in TT-Nb_2O_5) are components of effective catalysts for the ODH reactions, e.g., for dehydrogenative oxidation of propane to propylene [1–3]. Moreover, the semiconducting oxides V_2O_5 and Nb_2O_5 [4], owing to their electrical and magnetic properties, have found applications in the processes of producing novel materials used for example in medicine [5] or in lithium batteries as electrodes [6].

An ample scientific literature on the subject implies as well that vanadium–antimony oxide catalysts are active in the processes of utilizing such air pollutants as hydrogen sulfide, carbon monoxide, or the oxides of nitrogen [7, 8]. The catalysts based on the system V–Sb–O are also very active and selective in a technologically very important reaction of oxidative ammonolysis of propane to acrylonitrile [9].

The literature data suggest that attractive catalytic, electrical, or magnetic properties can be expected, too, in the case of hitherto unknown phases and compounds involving in their formation the oxides and compounds containing niobium, vanadium, and antimony. Before any investigations on application properties of these compounds and phases should be performed, it is necessary to conduct a primary research intended to find out in which systems they are formed, to establish the optimum conditions of their synthesis, and to determine their basic physicochemical properties.

The physicochemical properties of $SbVO_5$ and of the low-temperature polymorph of niobium(V) oxide

---

E. Filipek (✉) · M. Piz
Department of Inorganic and Analytical Chemistry, West Pomeranian University of Technology, Szczecin, al Piastów 42, 71-065 Szczecin, Poland
e-mail: elafil@zut.edu.pl

($T\text{-}Nb_2O_5$) are well known [10–15]. The compound $SbVO_5$, known only for the last 10 years, is formed in the system V–Sb–O among other ways through a reaction between V_2O_5 and $\alpha\text{-}Sb_2O_4$ in air at temperatures not exceeding 650 °C [10]. It is stable in air up to ca. 710 °C, and while further heated it decomposes—with an accompanying liberation of oxygen—to a rutile-type phase of the formula $Sb_{1-x}V_{1-x}O_4$, where $0.08 \leq x \leq 0.1$ [10–12]. The XRD characteristics of $SbVO_5$, its density, and its unit cell parameters are presented in the work [12]. It is known, too, that antimony and vanadium in $SbVO_5$ possess mainly the +5 oxidation state. The conducted EPR investigations have proved that the mass fraction of vanadium in the +4 oxidation state in $SbVO_5$ does not exceed 0.0002 [13].

The structure of $T\text{-}Nb_2O_5$—the other component of the investigated reacting mixtures—is well known [14, 15]. This low-temperature polymorph of niobium(V) oxide is built from pentagonal bipyramids and deformed octahedra, and a small amount of niobium ions occurs in structural voids surrounded by nine oxide ions. $T\text{-}Nb_2O_5$ crystallizes in the orthorhombic system [15]. It is known as well that at ca. 800 °C $T\text{-}Nb_2O_5$ undergoes a slow polymorphic transformation into $H\text{-}Nb_2O_5$, a phase crystallizing in the monoclinic system and melting at about 1480 °C [16].

It can be concluded from available literature, however, that the reactivity of $SbVO_5$ toward $T\text{-}Nb_2O_5$ has not been studied so far. The main aim of the present work was therefore investigating the mutual reactivity of $SbVO_5$ with $T\text{-}Nb_2O_5$ in the atmosphere of air. The results of these investigations will allow a statement whether any and what phases or compounds are formed through solid-state reactions between these two reacting substances in air; the acquired results will allow also a determination of the thermal stability and the basic physicochemical properties of the obtained phases or compounds.

Experimental procedure

The following reagents were used in our experiments:

$T\text{-}Nb_2O_5$ was a commercial pure product of Aldrich (Germany) and $SbVO_5$ was obtained by heating in air an equimolar mixture of V_2O_5 (p.a. product, POCh, Gliwice, Poland) with $\alpha\text{-}Sb_2O_4$ (obtained by heating a pure Sb_2O_3, Merck, Germany, in air) by a method described in the work [10].

For the experiments, 10 samples were prepared (Table 1), with their compositions representing the whole components concentrations range of the system under consideration.

The reagents weighed in suitable proportions were homogenized and calcined in the atmosphere of air at 600, 620, 650, 675, 690, and 720 °C in 24 or 48 h stages by a conventional method described in the papers [10, 17–19]. After each heating stage, an XRD examination was performed and selected samples were additionally subjected to a DTA/TG investigation.

The powder diffraction patterns of obtained samples were recorded with the aid of the diffractometer DRON-3 (Bourevestnik, Sankt Petersburg, Russia) using the radiation Cu Kα/Ni. Identification of phases was conducted on the basis of XRD characteristics contained in the PDF cards.

The DTA/TG investigations were performed by means of an F. Paulik–L. Paulik–L. Erdey derivatograph, product of MOM Budapest. The measurements were conducted in the atmosphere of air in the temperature range 20–1000 °C, at the DTA galvanometer sensitivity of 1/5 and a constant heating rate of 10 °C min^{-1}. The weighed amount of the samples was 500 mg in each case. The DTA/TG measurements of monophase samples in the temperature range 20–1500 °C were carried out with the use of an SDT 2960 apparatus (TA Instruments).

Table 1 Compositions of initial mixtures and phase analysis results for samples after selected stages of heating

No.	Composition/100 x		Phase composition of samples after their heating		
	$SbVO_5$	$T\text{-}Nb_2O_5$	Stage III 650 °C—24 h	Stage V 690 °C—24 h	Stage VI 720 °C—48 h
1	95.00	5.00	$T\text{-}Nb_2O_5$, $SbVO_5$, **Nb_2VSbO_{10}**	$SbVO_5$, **Nb_2VSbO_{10}**	$Sb_{0,92}V_{0,92}O_4$, **Nb_2VSbO_{10}**
2	90.00	10.00	$T\text{-}Nb_2O_5$, $SbVO_5$, **Nb_2VSbO_{10}**	$SbVO_5$, **Nb_2VSbO_{10}**	$Sb_{0,92}V_{0,92}O_4$, **Nb_2VSbO_{10}**
3	80.00	20.00	$T\text{-}Nb_2O_5$, $SbVO_5$, **Nb_2VSbO_{10}**	$SbVO_5$, **Nb_2VSbO_{10}**	$Sb_{0,92}V_{0,92}O_4$, **Nb_2VSbO_{10}**
4	70.00	30.00	$T\text{-}Nb_2O_5$, $SbVO_5$, **Nb_2VSbO_{10}**	$SbVO_5$, **Nb_2VSbO_{10}**	$Sb_{0,92}V_{0,92}O_4$, **Nb_2VSbO_{10}**
5	60.00	40.00	$T\text{-}Nb_2O_5$, $SbVO_5$	$SbVO_5$, **Nb_2VSbO_{10}**	$Sb_{0,92}V_{0,92}O_4$, **Nb_2VSbO_{10}**
6	50.00	50.00	$T\text{-}Nb_2O_5$, $SbVO_5$	$T\text{-}Nb_2O_5$, $SbVO_5$, **Nb_2VSbO_{10}**	**Nb_2VSbO_{10}**
7	40.00	60.00	$T\text{-}Nb_2O_5$, $SbVO_5$	$T\text{-}Nb_2O_5$, $SbVO_5$, **Nb_2VSbO_{10}**	**Nb_2VSbO_{10},** $T\text{-}Nb_2O_5$
8	30.00	70.00	$T\text{-}Nb_2O_5$, $SbVO_5$	$T\text{-}Nb_2O_5$, $SbVO_5$, **Nb_2VSbO_{10}**	**Nb_2VSbO_{10},** $T\text{-}Nb_2O_5$
9	20.00	80.00	$T\text{-}Nb_2O_5$, $SbVO_5$	$T\text{-}Nb_2O_5$, $SbVO_5$, **Nb_2VSbO_{10}**	**Nb_2VSbO_{10},** $T\text{-}Nb_2O_5$
10	10.00	90.00	$T\text{-}Nb_2O_5$, $SbVO_5$	$T\text{-}Nb_2O_5$, $SbVO_5$	$T\text{-}Nb_2O_5$

Selected samples were investigated also by means of an electron scanning microscope—SEM/WDX (JSM-1600, Jeol, Japan).

The densities of monophase samples containing only Nb_2VSbO_{10} were determined by degassing samples and hydrostatic weighing in pycnometric liquid (CCl_4) by the method described in the paper [20].

Results and discussion

The composition of initial mixtures of the reacting substances, i.e., $SbVO_5$ with T-Nb_2O_5, is presented in Table 1. Table 1 lists also the phase composition of the investigated samples after selected stages of heating them.

The phase analysis of all samples, performed after the first and the second stage of heating them, showed that the heating of the samples at 600 °C (24 h) and next at 620 °C (24 h) did not cause any change in their composition, and they still contained $SbVO_5$ with T-Nb_2O_5.

In the diffractograms of samples 1–4, i.e., the samples that contained in their initial mixtures the mole fraction $x = 0.0500$ to $x = 0.3000$ of T-Nb_2O_5 mixed with $SbVO_5$, taken after the third stage of heating (650 °C for 24 h), some new diffraction lines were present beside the XRD line sets characteristic for $SbVO_5$ and T-Nb_2O_5. These new lines could not possibly be ascribed to any XRD characteristics of the known compounds or phases formed in the systems Sb–V–O, Nb–V–O, and Sb–Nb–O [10, 21–29]. The fourth (675 °C—48 h) and the fifth (690 °C—24 h) stage of heating caused the samples that in their initial mixtures had comprised up to $x = 0.4000$ of T-Nb_2O_5 (mixed with $SbVO_5$) to be diphase and to contain only the new compound beside $SbVO_5$. Any presence of niobium(V) oxide was not detected in these samples any more. After these and the next (720 °C—48 h) heating cycles, the sample that had been initially an equimolar mixture of the title reacting substances was monophase and contained only the new compound. In the samples that represented the remaining concentration range of the investigated system $SbVO_5$–T-Nb_2O_5, i.e., samples comprising in their initial mixtures more than $x = 0.5000$ of T-Nb_2O_5, the new phase began to be formed not before the heating at 690 °C for 24 h. After a further heating stage (VI: 720 °C—48 h), these samples were diphase (except for sample 10), and beside the new compound they contained only T-Nb_2O_5. At this stage of our research, we assumed that the absence of any diffraction lines characteristic for the new compound, beside some lines of very large intensities belonging to the line set of T-Nb_2O_5, in the diffractogram of sample 10, can be due to a very small content of the new compound, i.e., a content below its limit of detection by XRD. It cannot be excluded, as well, that in this component concentration range of the investigated system—a range represented only by sample 10—a low-concentration solid solution with a T-Nb_2O_5-type structure is formed. In order to confirm this supposition, some separate investigations must be conducted that did not pertain to the subject of this paper, but we will perform them in the near future.

The phase composition of all investigated samples as well as our tracking of intensity changes of the diffraction lines characteristic for the new, hitherto unknown compound have indicated that in the reacting mixtures $SbVO_5$ + T-Nb_2O_5 a reaction occurs in accordance with the equation:

$$Nb_2O_{5(s)} + SbVO_{5(s)} = Nb_2VSbO_{10(s)} \qquad (1)$$

The new compound Nb_2SbVO_{10} has a brownish sandy sort of color.

Figure 1 presents a fragment of a diffractogram of: (a) an equimolar mixture of $SbVO_5$ with T-Nb_2O_5; (b) the compound Nb_2VSbO_{10} obtained from the above mixture.

It can be evidently concluded from Fig. 1 that the diffraction lines belonging to the set characteristic for the new compound Nb_2VSbO_{10} differ both as to their positions and as to their mutual intensity relations from the XRD line sets characteristic for the components of the initial equimolar mixture $SbVO_5$ + T-Nb_2O_5 [10, 12, 14].

Preliminary results of this part of our research were presented during 10th CCTA in Zakopane (Poland) in 2009 [30].

In the next stage of our research, the powder diffraction pattern of Nb_2VSbO_{10} (Cu Kα/Ni) was subjected to indexing by means of the program Powder, in order to find out in which crystallographic system this compound crystallizes and to calculate its unit cell parameters. The deviation of the reflection positions in the Nb_2VSbO_{10} diffractogram was established through XRD investigations

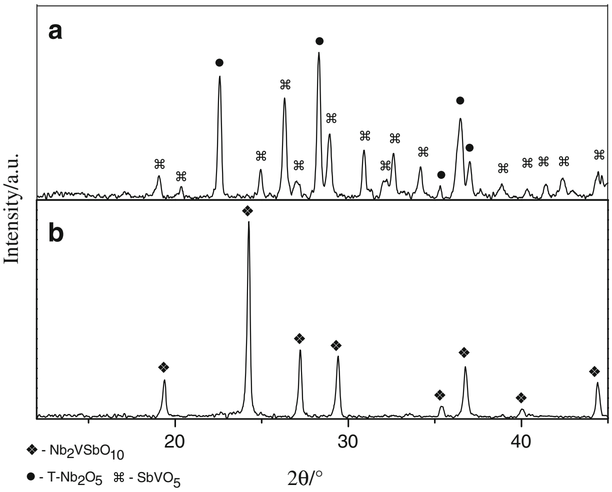

Fig. 1 X-ray diffraction patterns of: **a** initial mixture comprising $x = 0.5000$ of $SbVO_5$ + $x = 0.5000$ of T-Nb_2O_5 and **b** Nb_2VSbO_{10}

Table 2 Indexing results for the powder diffraction pattern of Nb_2VSbO_{10}

No.	Miller indices *hkl*	d_{exp}/nm	d_{calc}/nm	100 *I*
1	002	0.6124	0.6122	3
2	010	0.4584	0.4584	22
3	012	0.3672	0.3669	100
4	100	0.3282	0.3277	29
5	013	0.3043	0.3048	29
6	110	0.2663	0.2666	2
7	014	0.2541	0.2546	3
8	112	0.2448	0.2444	26
9	021	0.2255	0.2253	5
10	006	0.2043	0.2041	21
11	024	0.1833	0.1836	20
12	017	0.1636	0.1635	14
13	133	0.1314	0.1313	4
14	225	0.1172	0.1172	5

of a mixture of the new compound with an internal standard, i.e., with α-Al_2O_3. For the indexing, 14 reflections were selected from the 2θ range 4–90° (Table 2). An analysis of a series of obtained solutions, and especially a high value of FM as well as the calculated density value 4.67 g cm^{-3} very close to the experimentally measured density 4.68 ± 0.05 g cm^{-3}, have indicated that the new compound Nb_2VSbO_{10} crystallizes in the orthorhombic system. The calculated unit cell parameters are the following: $a = 0.328143$ nm, $b = 0.458946$ nm, $c = 1.22476$ nm, and $Z = 1$.

Investigations aiming at a determination of the thermal properties of the compound Nb_2VSbO_{10} were carried out by differential thermal analysis (DTA).

In the DTA curve of Nb_2VSbO_{10} up to 1000 °C under air, one distinct endothermic effect was recorded, beginning at about 880 °C (Fig. 2).

In order to explain the nature of the recorded DTA effect, a monophase sample containing Nb_2VSbO_{10} was subjected to additional investigations. For this purpose, the sample was heated for 3 h under air, at a temperature close to the maximum temperature of the DTA effect, i.e., at 920 °C. Next the sample was rapidly cooled to room temperature and analysed with respect to its phase composition.

A phase composition analysis of this sample, conducted on the base of its diffractogram (XRD), showed the presence of Nb_9VO_{25} [24] beside $Sb_{1-x}V_{1-x}O_4$ [21–23] as well as small amounts of vanadium(V) oxide. Having considered that under the conditions of the experiment, the sample underwent a partial melting and that among the identified phases only the compound Nb_9VO_{25} exists as a solid phase at 920 °C [29], we concluded that the endothermic effect recorded in the DTA curve is due to an incongruent melting of Nb_2VSbO_{10} in accordance with the peritectic reaction:

$$Nb_2VSbO_{10(s)} \leftrightarrow Nb_9VO_{25(s)} + \text{liquid} \qquad (2)$$

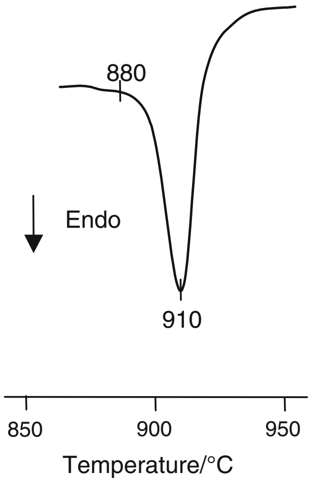

Fig. 2 DTA curve of Nb_2VSbO_{10} (sample 5 after heating at 720 °C—48 h)

Thus, the solid product of the new compound Nb_2VSbO_{10} melting at 880 °C is Nb_9VO_{25}, whereas $Sb_{1-x}V_{1-x}O_4$ and V_2O_5 are phases crystallizing from the liquid. Correctness of this statement is corroborated by the fact that in the diffractogram of Nb_2VSbO_{10} heated at 920 °C (3 h) and next rapidly cooled to room temperature only the XRD lines belonging to the sets characteristic for $Sb_{1-x}V_{1-x}O_4$ and V_2O_5 differed both as to their mutual intensity relations (I/I_0) and as to the corresponding interplanar distances (d) from the XRD characteristics of these phases contained by the PDF files Nos. 9-387 and 30-1412, 16-600. These differences are due to a very quick crystallization of these phases from the liquid during the rapid cooling of the investigated sample from 920 °C to ambient temperature.

In a further part of this work, the compound Nb_2VSbO_{10} and the samples obtained after heating this compound at 920 °C were subjected to investigations by scanning electron microscopy (SEM).

Figure 3 presents a SEM image of a monophase sample containing only the new compound Nb_2VSbO_{10}, whereas Fig. 4a and b shows SEM images of two selected fragments of a sample obtained after melting Nb_2VSbO_{10} at 920 °C.

The crystals of Nb_2VSbO_{10} visible in Fig. 3, with their shape of irregular polyhedra and their size ranging from 1 to several micrometers, differ considerably both as to the shape and as to the size from the crystals produced after melting Nb_2VSbO_{10} that exhibit three different kinds of the shape. In the images of the molten sample, apart from

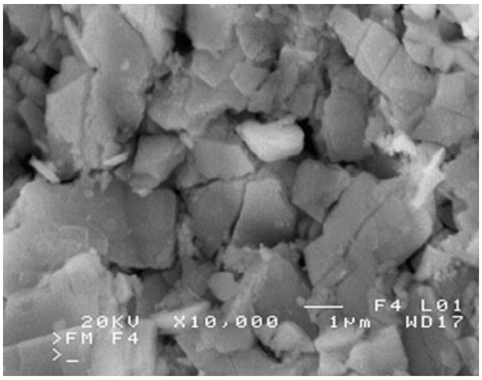

Fig. 3 SEM image of a monophase sample containing only the new compound Nb_2VSbO_{10}

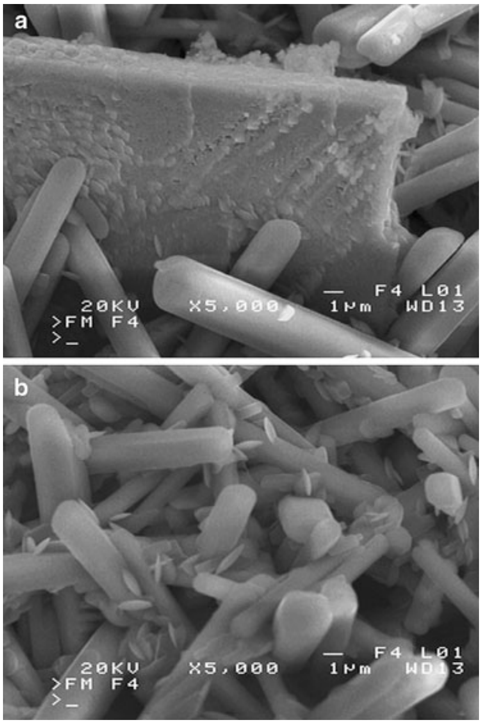

Fig. 4 SEM image of Nb_2VSbO_{10} heated at 920 °C **a** during 3 h (crystals of Nb_9VO_{25} and $Sb_{1-x}V_{1-x}O_4$) and **b** during 6 h (crystals of Nb_9VO_{25} shaped like "massive sticks")

crystals resembling "cuboidal plates" and very small—in comparison with them—"circular" crystals (of ca. 1 μm in diameter), some crystals can be clearly distinguished that have a shape of "massive sticks" and are several to a dozen or so micrometer long (Fig. 4a and b).

An X-ray microanalysis (SEM/EDX) of the content of niobium, vanadium, and antimony in the investigated samples was carried out in several points chosen within each sample on the biggest crystals whose one face was perpendicular to the electron beam. This chemical analysis of Nb_2VSbO_{10} crystals, performed pointwise, has proved the presence of the determined elements in the following averaged amounts (molar fractions): $x_{Nb} = 0.5054$, $x_V = 0.2464$, and $x_{Sb} = 0.2482$, in comparison with the contents calculated from the chemical formula of the compound: $x_{Nb} = 0.5000$, $x_V = 0.2500$, and $x_{Sb} = 0.2500$. Considering that the mean error of the above determination amounted to 0.003–0.008, we concluded that this analysis result is an additional corroboration of the proposed formula of the new compound. Moreover, an X-ray microanalysis (SEM/EDX) of the crystals shaped like "massive sticks" in the sample taken after heating Nb_2VSbO_{10} at 920 °C has shown that they contain niobium and vanadium in amounts corresponding to Nb_9VO_{25}, i.e., to the solid product of the incongruent melting of the new compound.

In the next stage of our research, two selected samples (that one which after the fifth heating stage—at 690 °C for 24 h—contained $SbVO_5$ with Nb_2VSbO_{10} as well as that one which after the sixth heating cycle—at 720 °C for 48 h—was a mixture of Nb_2VSbO_{10} and T-Nb_2O_5) were subjected to additional DTA/TG investigations (Figs. 5 and 6).

In the DTA curve of the mixture of $SbVO_5$ with Nb_2VSbO_{10} ($x = 0.20$ of T-Nb_2O_5 in the initial mixture of the reacting substances), as presented in Fig. 5, three endothermic effects were recorded up to 1000 °C.

The first effect with its onset temperature equal to 710 °C is due, according to literature data [10, 11, 13], to a thermal decomposition of $SbVO_5$. It is known that under air the compound $SbVO_5$ decomposes—with an accompanying liberation of oxygen—to a nonstoichiometric rutile-type phase, namely to $Sb_{0.92}V_{0.92}O_4$, that melts at ~820 °C. Thus, the second endothermic effect recorded in the DTA curve of the investigated sample is caused by a melting of $Sb_{0.92}V_{0.92}O_4$. The third thermal effect in this

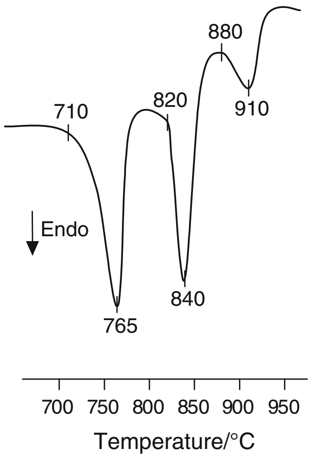

Fig. 5 DTA curve of a mixture of $SbVO_5$ with Nb_2VSbO_{10} (sample 2 after heating at 690 °C—24 h)

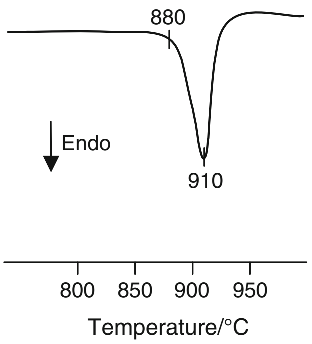

Fig. 6 DTA curve of a mixture of Nb_2VSbO_{10} with T-Nb_2O_5 (sample 7 after heating at 720 °C—48 h)

DTA curve with its onset temperature equal to 880 °C is due, according to our research and its results described in this article, to an incongruent melting of the new compound Nb_2VSbO_{10} that was present in the investigated mixture. An additional confirmation for the above interpretation of the first effect, i.e., that beginning at 710 °C, is the phase composition of those samples (comprising up to $x = 0.5000$ of T-Nb_2O_5 in the initial mixtures of the reacting substances) which after their heating in the sixth cycle at 720 °C were mixtures that contained the new compound Nb_2VSbO_{10} with $Sb_{0.92}V_{0.92}O_4$, or in other words—with the solid product yielded by decomposing $SbVO_5$. The heating of these samples at 720 °C, unlike the other stages of their roasting, was accompanied by mass losses that were associated with the liberation of oxygen during the decomposition of $SbVO_5$ present in the mixtures.

One endothermic effect beginning at 880 °C, recorded in the DTA curve (Fig. 6) of the sample that after the final heating cycle at 720 °C was a mixture of Nb_2VSbO_{10} with T-Nb_2O_5 ($x = 0.6000$ of T-Nb_2O_5) in the initial mixture of the reacting substances), is due, according to the results of these investigations, to a melting of Nb_2VSbO_{10}.

Conclusions

The research results acquired within this work entitle the following conclusions to be drawn:

1. The compound $SbVO_5$, known since a short time, reacts in solid state in air with T-Nb_2O_5 forming a new compound to which the formula Nb_2VSbO_{10} has been ascribed.
2. The new compound Nb_2VSbO_{10} has a brownish sandy sort of color and the density 4.67 g cm^{-3}. It is stable under air up to ~880 °C, and next it melts incongruently with an accompanying deposition of solid Nb_9VO_{25}.
3. Nb_2VSbO_{10} crystallizes in the orthorhombic system, and the calculated parameters of its unit cell that has the volume $V = 0.184448$ nm^3 are the following: $a = 0.328143$ nm, $b = 0.458946$ nm, $c = 1.22476$ nm. The number of the stoichiometric units of the compound per such unit cell amounts to 1.
4. The new compound Nb_2VSbO_{10} coexists with $SbVO_5$ in solid state under air up to 710 °C, and with T-Nb_2O_5—up to ~800 °C.

References

1. Barbieri F, Cauzzi D, Smet de F, Devillers M, Moggi P, Predieri G, Ruiz P. Mixed-oxide catalysts involing V, Nb, and Si obtained by non-hydrolytic sol–gel route: preparation and catalytic behaviour in oxydative dehydrogenation of propane. Catal Today. 2000;61:353–60.
2. Sarzi-Amade MS, Moggi P, Maione A, Ruiz P, Devillers M. The effect of sol–gel promoters on the characteristics of mixed V–Nb oxides and their catalytic properties in propane oxidative dehydrogenation. Appl Catal A. 2005;284:11–20.
3. Mimura Y, Ohyachi K, Matsuura I. Selective ammoxidation of propane over Sb–Nb–V oxide catalysts with α-Sb_2O_4 and rutile type structure. Stud Surf Sci Catal. 1998;121:69–74.
4. Amountza A, Riza F, Kasselouri V. Electrical conductivity of the ceramic compounds VNb_9O_{25} and VTa_9O_{25}. Mater Lett. 2003;57:2298–304.
5. Arkhipova EV, Zuev MG, Perelyaeva LA. Phase relations and spectral properties of new phases in Sc_2O_3–V_2O_5–Nb_2O_5–Ta_2O_5 system. J Alloy Compd. 2006;414:48–54.
6. Kumagai N, Ikenoya I, Ishiyama I, Tanno K. Electrochemical and structural characteristics of niobium vanadium oxide electrodes in a secondary lithium battery. Solid State Ionics. 1988;28–30:862–7.
7. Kuo-Tseng L, Ni-Shen S. Catalytic oxidation of hydrogen sulfide to sulfur on vanadium antimonate. Ind Eng Chem Res. 1997;36:1480–4.
8. Dae-Won P, Byung-Kook P, Dae-Kwang P, Hee-Chul W. Vanadium–antimony mixed oxide catalysts for the selective oxidation of H_2S containing excess water and ammonia. Appl Catal A. 2002;223:215–24.
9. Centi G, Perathoner S, Trifiro F. V–Sb-oxide catalysts for the ammoxidation of propane. Appl Catal A. 1997;157:143–72.
10. Filipek E. Phase equilibria in the V_2O_5–Sb_2O_4. J Therm Anal Calorim. 1999;56:159–65.
11. Typek J, Filipek E, Maryniak M, Guskos N. Magnetic resonance study of $SbVO_5$ thermal decomposition products. Mater Sci Pol. 2005;23:1047–54.
12. Filipek E. Homogeneity area of MoO_3 solid solution in $SbVO_5$. Solid State Sci. 2006;8:577–88.
13. Typek J, Guskos N, Buchowski D, Wabia M, Filipek E. EPR study of monomeric and dimeric vanadyl ions in $SbVO_5$. Radiat Eff Defect Solid. 2002;157:1093–9.
14. Tamura S, Kato K, Goto M. Single crystals of T-Nb_2O_5 obtained by slow cooling method under high pressures. Z Anorg Allg Chem. 1974;410:313–5.
15. Holser WT. Compounds with α-U_3O_8-type structure. Acta Cryst. 1956;9:196.
16. Kato K. Structure refinement of H-Nb_2O_5. Acta Cryst. 1976;B32:764–7.

17. Tabero P. Thermal expansion of $MNbO_4$ phases where M = Al, Cr, Fe,Ga. J Therm Anal Calorim. 2007;88:269–72.
18. Bosacka M, Blonska-Tabero A. Reinvestigation of system CdO–V_2O_5 in the solid state. J Therm Anal Calorim. 2008;93:811–5.
19. Tomaszewicz E, Typek J, Kaczmarek SM. Synthesis, characterization and thermal bahaviour of new copper and rare-earth metal tungstates. J Therm Anal Calorim. 2009;98:409–21.
20. Kluz Z, Waclawska I. Precise determination of powder density. Roczniki Chem. 1974;49:839–41.
21. Berry FJ, Brett ME, Patterson WR. Structural investigations of the vanadium–antimony–oxygen system. J Chem Soc Dalton Trans. 1983;13–7.
22. Berry FJ, Brett ME, Patterson WR. Vanadium antimonate. J Chem Soc Dalton Trans. 1983;9–12.
23. Birchall T, Sleight AW. Oxidation states in $VSbO_4$. Inorg Chem. 1976;15:868–70.
24. Casais MT, Gutierrez-Puebla E, Monge MA, Rasines I, Ruiz-Valero C. VM_9O_{25} (M = Nb, Ta), a combination of tetrahedral VO_4 and octahedral MO_6 units. J Solid State Chem. 1993;102: 261–6.
25. Yamaguchi O, Mukaida Y, Shigeta H. New compounds in the $Nb_2O_5(Ta_2O_5)$–V_2O_5 systems. Z Anorg Allg Chem. 1989;574: 235–8.
26. Amarilla J, Casal B, Ruiz-Hitzky E. MVO_5 (M = Nb, Ta) mixed oxides: sol–gel synthesis, structural and thermal characterization and electrochemical Li^+ insertion. J Mater Chem. 1996;6: 1005–11.
27. Popolitov VI, Ivanova LA, Stephanovitch SYu, Chetchkin VV, Lobachev AN, Venevtsev YuN. Ferroelectrics ABO_4: synthesis of single crystals and ceramics; dielectric and nonlinear optical properties. Ferroelectrics. 1974;8:519–20.
28. Blanton TN, Barnes CL, Eichorst DJ. X-ray powder diffraction data for cubic $Sb_3Nb_3O_{13}$. Powder Diffr. 1993;8:188–90.
29. Waring JL, Roth RS. Phase equilibria in the system vanadium oxide–niobium oxide. J Res NBS A Phys Ch. 1965;69A:119–29.
30. Filipek E, Piz M. The reactivity in air of $SbVO_5$ towards T-Nb_2O_5 in the solid state. In: Proc 10th conference on calorimetry and thermal analysis joint with 2nd Czech–Hungarian–Polish–Slovakian thermoanalytical conference; 2009. p. 41.