# Phase Equilibria in the V–Ni–Sb System

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**Abstract**—Phase relations in the V–Ni–Sb system have been studied by x-ray diffraction, and the 1070-K section of its phase diagram has been constructed in the region 0–67 at % Sb. The VSb phase (NiAs structure) is shown to have a homogeneity range:  $V_{1.04-1.00}Sb_{0.96-1.00}$  (a = 0.4274(1)–0.4266(2) nm, c = 0.5464(2)–0.5448(4) nm). The binary antimonides with the NiAs structure dissolve the third component. V solubility in NiSb is within 3.5 at %. Ni dissolution in VSb is accompanied by partial Ni substitution for V and Ni incorporation into position 2*d* of space group  $P6_3/mmc$ , up to the composition  $V_{0.84}Ni_{0.32}Sb_{0.84}$  (a = 0.42187(6) nm, c = 0.54000(9) nm).

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### **INTRODUCTION**

Earlier phase-diagram studies of the V-Ni-Sb system were concentrated on the formation of compounds. The VNiSb compound (MgAgAs structure type, sp. gr. F43m, a = 0.5785 nm [1]) was shown to form at 870 K. Using high-pressure synthesis, Noda et al. [2] obtained a compound which, most likely, had an Ni<sub>2</sub>In-type structure. From the unit-cell volume of that compound, its composition was inferred to be  $V_{0.6}$ NiSb (sp. gr.  $P6_3/mmc$ , a = 0.4217 nm, c = 0.5398 nm). The structure of the VNi<sub>0.26</sub>Sb phase (sp. gr.  $P6_3/mmc$ , a =0.42527(5) nm, c = 0.54443(6) nm) was determined by single-crystal x-ray diffraction (XRD) and was shown to be related to the Ni<sub>2</sub>In type [4]. In this paper, we report a systematic study of the phase equilibria in the V-Ni-Sb system and the 1070-K section of its phase diagram.

## **EXPERIMENTAL**

The V–Ni–Sb system was studied at 1070 K in the composition region 0–67 at % Sb. Samples for this investigation were prepared from high-purity (99.9+%) metal powders. After pressing, the green compacts were sintered at 1070 K for 200 h in evacuated silica ampules. Next, the samples were ground and sintered at the same temperature for an additional 800 h. At low Sb concentrations, the sintered samples were arc-melted and then homogenized by annealing at 1070 for 800 h, followed by quenching in cold water without breaking the vacuum. In our studies, we used only those samples which differed in weight from the starting mixture by no more than 2%.

The phase composition of the samples was determined by powder XRD with a DRON-3M diffractometer (Cu $K_{\alpha}$  radiation) and Debye–Scherrer camera (CrK radiation). Intensity data for structural analysis were collected on the DRON-3M (Cu $K_{\alpha}$  radiation, step size of 0.05°, counting time of 10 s per data point). All crystallographic calculations were made using the CSD software package [5].

#### **RESULTS AND DISCUSSION**

In the V–Ni system, we obtained all the binary compounds reported earlier to exist at 1070 K:  $V_3Ni$  (Cr<sub>3</sub>Si structure type),  $\sigma$ -VNi (CrFe) with an appreciable homogeneity range, VNi<sub>2</sub> (MoPt<sub>2</sub>), and VNi<sub>3</sub> (TiAl<sub>3</sub>). Their lattice parameters are listed in Table 1.

In the Ni–Sb system at 1070 K, we obtained the earlier reported compounds  $Ni_5Sb_2$  ( $Ni_5Sb_2$ ) and NiSb(NiAs). According to our results, the homogeneity range of the latter compound can be represented by  $Ni_{1.09-1.00}Sb_{0.91-1.00}$  (Fig. 1a) and is slightly narrower than that in the phase diagram presented in [6].

In the V–Sb system, we obtained the earlier reported compounds V<sub>3</sub>Sb (Cr<sub>3</sub>Si), V<sub>3</sub>Sb<sub>2</sub> (Fe<sub>3</sub>Sn<sub>2</sub>), V<sub>5</sub>Sb<sub>4</sub> (Ti<sub>5</sub>Te<sub>4</sub>) (by sintering an elemental powder mixture at 1070 K three times), VSb (NiAs), and VSb<sub>2</sub> (CuAl<sub>2</sub>). Since no V–Sb phase diagram has been reported to date, we studied this system in detail. In the composition range 42–56 at % Sb, samples were prepared at 2 at % Sb intervals by sintering at 1070 K in three steps. Only VSb was found to have a homogeneity range. From the composition dependences of its lattice parameters (Fig. 1b), the VSb phase was inferred to exist in the composition range  $V_{1.04-1.00}Sb_{0.96-1.00}$ . The lattice parameters of all the phases in the V–Sb system are given in Table 1.

The 1070-K section of the V–Ni–Sb phase diagram (0-67 at % Sb), inferred from the present XRD results for 80 samples, is displayed in Fig. 2.

| Tał | ole 1. | Crystal | data for | phases of | f the ` | V–Ni–Sb system |
|-----|--------|---------|----------|-----------|---------|----------------|
|-----|--------|---------|----------|-----------|---------|----------------|

| Phase  | Structure<br>type               | Sp. gr.                              | <i>a</i> , Å                              | b, Å  | <i>c</i> , Å                                | Source                 |
|--|---------------------------------|--------------------------------------|---|---|---|------------------------|
| Sb   | α-As                            | $R\bar{3}m$                          | 4.3084<br>4.309(1)                        |   | 11.247<br>11.286(4)                         | [7]<br>This work       |
| Ni   | Cu                              |                                      | 3.5238                                    |   |   | [7]                    |
| N; Sh*   |                                 |                                      | 3.5231(8)                                 |   |   | This work              |
| $N_{0.90} SU_{0.10}$   |                                 |                                      | 3.392                                     |   |   | [8]                    |
| $N_{0.92}Sb_{0.08}$  |                                 |                                      | 3.588                                     |   |   | [8]                    |
| $Ni_{0.92}Sb_{0.08}^{+}$   |                                 | Fm3m                                 | 3.596(3)                                  |   |   | This work              |
| $Ni_{0.92}V_{0.08}^{*}$  |                                 |                                      | 3.539                                     |   |   | [8]                    |
| Ni <sub>0.84</sub> V <sup>*</sup> <sub>0.16</sub>  |                                 |                                      | 3.5465(4)                                 |   |   | This work              |
| Ni <sub>0.86</sub> V <sub>0.10</sub> Sb <sub>0.04</sub>  |                                 |                                      | 3.5557(3)                                 |   |   | This work              |
| V  | α-Fe                            | Im3m                                 | 3.0240                                    |   |   | [7]                    |
| $V_{0.85} Ni_{0.15}^*$   |                                 |                                      | 2.9900                                    |   |   | [8]                    |
| $V_{0.93}Ni_{0.07}^*$  |                                 |                                      | 3.0134(3)                                 |   |   | This work              |
| Ni <sub>5</sub> Sb <sub>2</sub>  |                                 |                                      | 12.9458                                   | 5.4271  | 11.4568                                     | [8]                    |
| Ni <sub>5.0-4.4</sub> Sb <sub>2.0-2.6</sub>  | Ni <sub>5</sub> Sb <sub>2</sub> | <i>C</i> 2                           | 12.896(7)–12.9743(6)                      | $\beta = 151./1^{\circ}$<br>5.425(5)-5.427(2)   | 11.4432(2) -<br>11.4300(6)                  | This work              |
|  |                                 |                                      |   | $\beta = 151.57(9)^{\circ} - 151.61(1)^{\circ}$ | 11.4300(0)                                  |                        |
| $\begin{array}{l} Ni_{1.12-0.94}Sb_{0.88-1.06}\\ Ni_{1.09-1.00}Sb_{0.91-1.00} \end{array}$   | NiAs                            | P6 <sub>3</sub> /mmc                 | 4.032–3.910<br>4.015(1)–3.9376(4)         |   | 5.200–5.130<br>5.1750(2)–<br>5.1430(6)      | This work<br>This work |
| $Ni_{1.00}V_{0.07}Sb_{0.93}^{*}$   |                                 |                                      | 3.9348(5)                                 |   | 5.131(2)                                    | This work              |
| Ni <sub>0.93</sub> V <sub>0.07</sub> Sb <sup>*</sup> <sub>1.00</sub>   |                                 |                                      | 3.9398(4)                                 |   | 5.1408(6)                                   | This work              |
| VNi <sub>3</sub>   |                                 |                                      | 3.540                                     |   | 7.220                                       | [8]                    |
| <u>V<sub>1.0-1.4</sub>Ni<sub>3.0-2.6</sub></u>   | TiAl <sub>3</sub>               | I4/mmm                               | 3.537(1)-3.547(2)                         | 7 ( 4 1   | 7.222(4)-7.248(6)                           | This work              |
| $V_{10}$ $V$ | MoPt <sub>2</sub>               | Immm                                 | 2.559<br>2.529(1)-2.554(3)                | 7.641 /.641 /.661(4)                            | 3.549<br>3.555(3) - 3.537(2)                | [8]<br>This work       |
| $\frac{1.0-1.1}{\sigma-VNi: (V_{1,2}Ni_{0.8})}$  |                                 |                                      | 8.955                                     |   | 4.625                                       | [8]                    |
| $(V_{1.3}Ni_{0.7})$  | CrFe                            | $P4_2/mnm$                           | 9.004                                     |   | 4.663                                       | [8]                    |
| $\frac{\mathbf{v}_{1.03-1.42} \mathbf{N}_{0.97-0.58}}{\mathbf{V}_{2} \mathbf{N}_{1}}$  | CroSi                           | Pm3n                                 | <u>8.888(3)</u> –9.039(4)<br><u>4.710</u> |   | 4.557(4)-4.099(5)                           | 1111S WORK<br>[8]      |
| V 31 VI  | 01351                           | 1 m3n                                | 4.706(2)                                  |   |   | This work              |
| V <sub>3</sub> Sb  | Cr <sub>3</sub> Si              | Pm3n                                 | 4.932                                     |   |   | [8]<br>This work       |
| V Sh   | Fe Sn                           | <u> </u>                             | 5 551                                     |   | 20.35                                       | 11115 WOIK<br>[8]      |
| v 3502   | 1035112                         | K S M                                | 5 557(3)                                  |   | 20.35                                       | This work              |
| $\overline{V_5Sb_4}$   | Ti₅Te₄                          | I4/m                                 | 9.81                                      |   | 3.50  | [8]                    |
|  | 5 4                             |                                      | 9.802(2)                                  |   | 3.502(4)                                    | This work              |
| VSb  | NiAs                            | <i>P</i> 6 <sub>3</sub> / <i>mmc</i> | 4.285                                     |   | 5.440                                       | [8]                    |
| $V_{1.04-1.00}Sb_{0.96-1.00}$  |                                 |                                      | 4.2/4(1)-4.266(2)<br>4.274(1)-4.2128(4)   |   | 5.464(2) - 5.448(4)<br>5 464(2)-            | This work              |
| 1.04-0.901 10-0.140 00.96  |                                 |                                      |   |   | 5.4033(8)                                   | THIS WORK              |
| $\begin{array}{l} V_{1.0-0.88}Ni_{0-0.12}Sb_{1.00} \\ V_{1.00-0.84}Ni_{0-0.12}Sb_{1.00-0.84} \end{array}$  |                                 |                                      | 4.266(2)-4.244(2)<br>4.274(1)-4.2187(6)   |   | 5.442(4)-5.366(4)<br>5.464(2)-<br>5.4000(9) | This work<br>This work |
| VSb <sub>2</sub>   | CuAl <sub>2</sub>               | I4/mcm                               | 6.5541                                    |   | 5.6365                                      | [8]                    |
| $V_{0.96}Sb_2$   |                                 |                                      | 6.5528(2)                                 |   | 5.8071(3)                                   | [9]                    |
| <u>v1.00-0.96502</u><br>VNiSh  | ΜσΔσΔο                          | F 1 2m                               | 5 860(5)                                  |   | 5.050(1)-5.044(1)                           | [1]                    |
| VocNiSh  | NiaIn                           | $P6_2/mmc$                           | 4.217                                     |   | 5.398                                       |                        |
| VNi <sub>0.26</sub> Sb   | Ni <sub>2</sub> In              | $P6_3/mmc$                           | 4.2527                                    |   | 5.443                                       | [4]                    |

\* Limit of the solid solution.



Fig. 1. Composition dependences of lattice parameters for (a) NiSb and (b) VSb; (1) single- and (2) two-phase samples.



Fig. 2. 1070-K section of the V-Ni-Sb phase diagram in the region 0-67 at % Sb: (1) single-, (2) two-, and (3) three-phase samples.

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| Composition   | NiV <sub>0.07</sub> Sb <sub>0.93</sub>      | Ni <sub>0.93</sub> V <sub>0.07</sub> Sb <sub>1.00</sub> |  |  |
|---|---|---|--|--|
| <i>a</i> , Å  | 3.9333(5)                                   | 3.9398(4)   |  |  |
| <i>c</i> , Å  | 5.1402(6)                                   | 5.1408(6)   |  |  |
| <i>V</i> , Å <sup>3</sup>   | 68.87(2)                                    | 69.10(2)  |  |  |
| Calculated density, g/cm <sup>3</sup>   | 8.458(3)                                    | 8.646(2)  |  |  |
| Diffractometer  | DRON-3M                                     |   |  |  |
| Radiation, wavelength   | $Cu, \lambda = 1.54185 \text{ Å}$           |   |  |  |
| Absorption coefficient, cm <sup>-1</sup>  | 1642.33                                     | 1739.56   |  |  |
| [001] texturing factor  | 0.7400                                      | 3.7(4)  |  |  |
| Refinement parameters   |   | 3   |  |  |
| Refinement procedure  | Full profile                                |   |  |  |
| $2\theta_{\text{max}}$ , deg; $\sin\theta_{\text{max}}/\lambda$ , Å <sup>-1</sup> | 97.89; 0.489                                | 97.89; 0.489  |  |  |
| Atomic positions  | 2   |   |  |  |
| Site composition; thermal parameter, $\text{\AA}^2$                               | Ni in $2a; B = 0.7(4)$                      | 0.90(3)Ni + $0.10(3)$ V in $2a$ ; $B = 0.7(4)$          |  |  |
|   | 0.93(5)Sb + $0.07(5)$ V in 2c; $B = 0.5(3)$ | Sb in $2c$ ; $B = 1.0(2)$                               |  |  |
| $R_I; R_p$  | 0.0249; 0.1545                              | 0.0650; 0.1146  |  |  |

**Table 2.** Crystal data for NiV<sub>0.07</sub>Sb<sub>0.93</sub> and Ni<sub>0.93</sub>V<sub>0.07</sub>Sb<sub>1.00</sub> (sp. gr.  $P6_3/mmc$ , Z = 2), intensity data collection conditions, and refinement statistics

It can be seen from Table 1 that the lattice parameters of  $Ni_5Sb_2$  vary across its homogeneity range. The variation is somewhat larger in three-component samples, attesting to a slight solubility of vanadium in  $Ni_5Sb_2$ . Since the samples containing 4 at % V were two- or three-phase, we conclude that the solubility of vanadium in  $Ni_5Sb_2$  is within 4 at %.

In three-component samples, the lattice parameters of the NiSb phase  $(Ni_{1-x}Sb_x)$  were slightly larger and the lattice parameters of the VSb phase  $(V_{1-x}Sb_x)$  were

slightly smaller than those of the respective binary compounds (Table 1), which also attests to dissolution of the third component (V and Ni, respectively).

To determine vanadium solubility in NiSb, we prepared samples containing 5 or 10 at % V and 44, 46, or 51 at % Sb. The samples containing 5 at % V were nearly single-phase (trace levels of additional phases). At a vanadium content of 10 at %, the samples were two- or three-phase. The solubility limit was determined by refining the crystal structure of NiSb (NiAs type) from powder XRD data for the samples contain-



**Fig. 3.** Composition dependences of lattice parameters for solid solutions of Ni in VSb along the (a)  $V_{1.04-x}Ni_xSb_{0.96}$ , (b)  $V_{1.00-x}Ni_xSb_{1.00}$ , and (c)  $V_{1.0-0.5x}Ni_xSb_{1.0-0.5x}$  lines: (1) single-, (2) two-, and (3) three-phase samples.

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Fig. 4. Relationships between the hcp (Mg type), NiAs, and Ni<sub>2</sub>In structures.

ing 5 at % V (nominal composition) and 44 or 46 at % Sb. The results (Table 2) indicate that NiSb dissolves no more than 3.5 at % V.

We also evaluated nickel solubility in VSb. Since VSb has a narrow homogeneity range ( $V_{1.04-1.00}Sb_{0.96-1.00}$ ), we prepared samples containing 42, 44, 46, 48, and 50 at % Sb. Ni solubility was determined from the composition dependences of lattice parameters along the  $V_{1.04-x}Ni_xSb_{0.96}$  and  $V_{1.00-x}Ni_xSb_{1.00}$  lines (Figs. 3a, 3b). As seen, the solubility limits are 7 and 6 at % Ni at Sb contents of 48 and 50 at %, respectively. The lattice parameters at these limits of the solid solution are given in Table 1.

XRD examination of three-component samples showed that the  $V_{0.45}Ni_{0.12}Sb_{0.43}$  and  $V_{0.43}Ni_{0.15}Sb_{0.42}$ samples were nearly single-phase and consisted of the VSb phase. In a recent study, Lewis et al. [4] determined the crystal structure of the VNi<sub>0.26</sub>Sb phase by single-crystal XRD. According to their results, position 2d (sp. gr. P6<sub>3</sub>/mmc) in this phase is occupied by nickel atoms only partially, i.e., it has a defect Ni<sub>2</sub>In structure. The NiAs and Ni<sub>2</sub>In structures types can be derived from the hcp structure (Mg type): filling the octahedral interstices of the hcp structure gives the NiAs type, and filling the double tetrahedral interstices leads to the  $Ni_2In$  type (Fig. 4). This led us to assume that nickel atoms can be incorporated into the double tetrahedral interstices of the VSb (NiAs) structure to form  $V_{1-0.5x}Ni_xSb_{1-0.5x}$  solid solutions. To validate this assumption, we determined the crystal structures of the solutions the single-phase solid in samples  $V_{0.45}Ni_{0.12}Sb_{0.43}$  and  $V_{0.43}Ni_{0.15}Sb_{0.42}$  by powder XRD, using the Ni<sub>2</sub>In structure as the input model (Table 3). The results demonstrate that positions 2a (000) and 2c $(1/3 \ 2/3 \ 1/4)$  are occupied by the V and Sb atoms and that position 2d (1/3 2/3 3/4) in the Ni<sub>2</sub>In structure is partially occupied by the Ni atoms. Therefore, nickel incorporation into the VSb phase (NiAs) leads to the formation of a defect Ni<sub>2</sub>In structure. The composition dependences of lattice parameters for the

**Table 3.** Crystal data for  $V_{0.90}Ni_{0.24}Sb_{0.86}$  and  $V_{0.86}Ni_{0.30}Sb_{0.84}$  (sp. gr.  $P6_3/mmc$ , Z = 2), intensity data collection conditions, and refinement statistics

| Composition   | V <sub>0.90</sub> Ni <sub>0.24</sub> Sb <sub>0.86</sub> | $V_{0.86}Ni_{0.30}Sb_{0.84}$  |  |  |  |
|---|---|-------------------------------|--|--|--|
| <i>a</i> , Å  | 4.2380(4)   | 4.2134(6)                     |  |  |  |
| <i>c</i> , Å  | 5.4467(6)   | 5.4225(8)                     |  |  |  |
| <i>V</i> , Å <sup>3</sup>   | 84.72(2)  | 83.31(6)                      |  |  |  |
| Calculated density, g/cm <sup>3</sup>   | 7.375(2)  | 7.272(2)                      |  |  |  |
| Diffractometer  | DRON-3M   |                               |  |  |  |
| Radiation, wavelength   | Cu, $\lambda = 1.54185 \text{ Å}$                       |                               |  |  |  |
| Absorption coefficient, cm <sup>-1</sup>  | 1542.47   | 1720.44                       |  |  |  |
| Texturing factor  | [1 0 0] 1.71  | [0 0 1] 0.4212                |  |  |  |
| Refinement parameters   |   | 1                             |  |  |  |
| Refinement procedure  | Full profile  |                               |  |  |  |
| $2\theta_{\text{max}}$ , deg; $\sin\theta_{\text{max}}/\lambda$ , Å <sup>-1</sup> | 110.77; 0.534   | 111.45; 0.536                 |  |  |  |
| Atomic positions  | 4   |                               |  |  |  |
| Site composition; thermal parameter, $Å^2$  | V in $2a; B = 2.6(5)$                                   | V in $2a; B = 3.7(5)$         |  |  |  |
|   | Sb in $2c; B = 2.7(3)$                                  | Sb in $2c$ ; $B = 3.3(9)$     |  |  |  |
|   | 0.27(2)Ni in 2d; $B = 1.6(12)$                          | 0.35(4)Ni in 2d; $B = 4.4(2)$ |  |  |  |
| $R_I; R_p$  | 0.0572; 0.2603  | 0.0573; 0.2728                |  |  |  |

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 $V_{1-0.5x}Ni_xSb_{1-0.5x}$  solid-solution system (Fig. 3c) indicate that the solid solution extends to the composition  $V_{0.78}Ni_{0.44}Sb_{0.78}$ . Thus, Ni dissolution in VSb involves both Ni substitution for V and partial incorporation of Ni atoms into position 2*d*. The VNi<sub>0.26</sub>Sb phase reported by Lewis et al. [4] seems to belong to the solid-solution system in question.

In our preparations at 1070 K, we failed to obtain (by sintering in several steps or arc melting) the VNiSb phase (MgAgAs structure type) [1] and the V<sub>0.6</sub>NiSb phase (presumably, Ni<sub>2</sub>In structure) [3] (the composition was inferred from the unit-cell volume). It may be that V<sub>0.6</sub>NiSb is formed under other conditions or has a different composition: its lattice parameters (a =0.4217 nm, c = 0.5398 nm [3]) differ very little from those at the limit of the V<sub>1-0.5x</sub>Ni<sub>x</sub>Sb<sub>1-0.5x</sub> solid solution (Table 1). Thus, we identified no ternary compounds in the V–Ni–Sb system at 1070 K.

The absence of ternary compounds in the V–Ni–Sb system and the formation of solid solutions based on binary compounds seem to be associated with both the low reactivity of antimony and the small difference in electronegativity and atomic size between the constituent transition metals ( $r_{\rm V} = 0.1321$  nm,  $r_{\rm Ni} = 0.1246$  nm;  $\chi_{\rm V}$  = 1.63,  $\chi_{\rm Ni}$  = 1.75 [7]). The phase relations in the V-Ni-Sb system are similar to those in systems with the other iron group metals or manganese. In particular, the V-Mn-Sb system contains substitutional solid solutions based on most of the binary antimonides [10], and those based on MnSb (NiAs) exist in a rather broad composition range. The systems V-Fe(Co)-Sb contain the compounds VFeSb and VCoSb with the MgAgAs structure, which undergo a high-pressure transition to the Ni<sub>2</sub>In structure [2]. The phase relations in the V-Ni-P(As,Sb) systems illustrate that, in systems formed by two transition metals and a Group V-A p element, the number of compound decreases in going from phosphorus (three compounds [11]) to arsenic (one compound [8]) and to antimony (no compounds). This is due to the reduction in the electronegativity of the p element and the increase in its atomic radius ( $r_{\rm P}$  = 0.110 nm,  $r_{As} = 0.125$  nm,  $r_{Sb} = 0.182$  nm;  $\chi_P = 2.10$ ,  $\chi_{As} = 2.08$ ,  $\chi_{Sb} = 1.82$  [7]). It is, therefore, reasonable to expect that the as yet unexplored system V–Ni–Bi also contains no compounds.

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