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Nuclear Instruments and Methods in Physics Research B xxx (2002) xxx–xxx

NIM B
 Beam Interactions
 with Materials & Atoms

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Development of an Au–Dy–Si liquid alloy ion source for focussed ion beam implantation

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Received 27 December 2001; received in revised form 25 March 2002

Abstract

A liquid metal ion source (LMIS) based on the Au_{78.2}Dy₈Si_{13.8} alloy was developed for focussed ion beam implantation of Dy ions. The mass spectrum of the LMIS shows the presence of Si²⁺, Si⁺, Dy³⁺, Dy²⁺, Au²⁺, Au⁺, Au₃²⁺ and Au₂⁺ ions. The Au–Dy–Si LMIS shows high stability and long time operational capacity. A simple calculational technique for eutectic compositions of ternary alloys for LMIS is suggested on the base of binary phase diagrams of the used elements. © 2002 Published by Elsevier Science B.V.

Keywords: Focused ion beam; Alloy liquid metal ion source; Ternary alloy; Dysprosium ions

1. Introduction

Focusing ion beam (FIB) systems are used for maskless ion implantation in electronic structure fabrication, microfabrication by microetching and deposition and ion beam lithography [1–3]. The increasing application of FIB implantation results in a need of new ions of different chemical elements in liquid metal ion sources (LMIS). The fast growing interest towards implantation of magnetic ions [4–6] in semiconductors increases the demand for alloys of such elements suitable for long time operation in LMIS. Dysprosium, possessing a large magnetic moment, represents such an ele-

ment. However, its utilization in LMIS is complicated due to its high melting temperature of 1409 °C and high vapor pressure of 76.2 Pa at this temperature. One of the very promising systems is the ternary Au–Si system with a third element, in this case Dy. The basic advantages of such alloys are low melting temperature, high wetting characteristics of the needle surface and the reservoir and high resistance to oxidation [7]. The most popular alloy on this base is Au₇₀Si₁₅Be₁₅ widely used in FIB implantation of A_{III}B_V semiconductors [3,8].

In this paper a simple calculational technique of ternary alloys for LMIS and results of investigations of an Au–Dy–Si liquid alloy ion source for focussed ion beam implantation are presented.

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44 2. Experiment

45 The alloy $\text{Au}_{78.2}\text{Dy}_8\text{Si}_{13.8}$ (atomic%) was pro-
 46 duced by melting of a preliminary pressed mixture
 47 of Au, Dy and Si pieces in an arc discharge furnace
 48 in Argon atmosphere. In order to increase the
 49 homogeneity, the ingot was turned over with
 50 subsequent melting. In Fig. 1 an EDX spectrum of
 51 the fabricated alloy is shown. In the spectrum lines
 52 corresponding to all three elements can be ob-
 53 served, which gives evidence of formation of a
 54 ternary alloy. Then, the alloy was placed in a
 55 graphite crucible for the filling of the LMIS.

56 A hairpin construction of the emitter with a
 57 spiral reservoir, often used in LMIS, was fabri-
 58 cated of 0.2 mm Tungsten wire. Cleaning and
 59 needle fabrication was conducted according to the
 60 technique described in [3]. An overall view of a
 61 filled LMIS is presented in Fig. 2. To prevent
 62 wetting of the heating wires by the melted alloy the
 63 wires were covered by ceramic glue. Filling of
 64 LMIS was performed by dipping it into the crucible
 65 with the melted Au–Dy–Si alloy in a separ-
 66 ated vacuum system “emitter maker” at a
 67 pressure of $<10^{-5}$ Pa. The wetting of Tungsten
 68 and the filling of the reservoir was performed
 69 without any problems as in the case of Au–Si–Be
 70 alloys.

71 After preliminary testing, the ion source was
 72 mounted in an EIKO-100 FIB system used for
 73 analysis of ion beam mass spectra by means of an

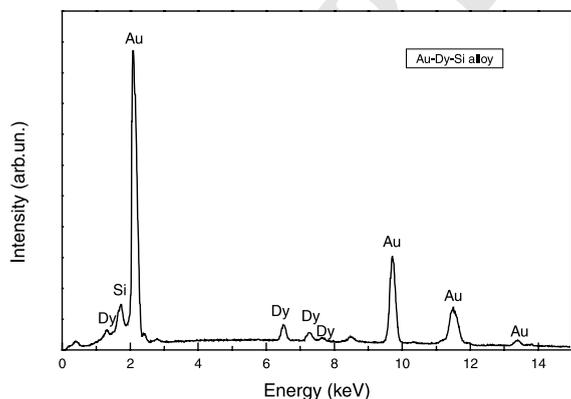


Fig. 1. EDX spectrum of the employed Au–Dy–Si alloy.

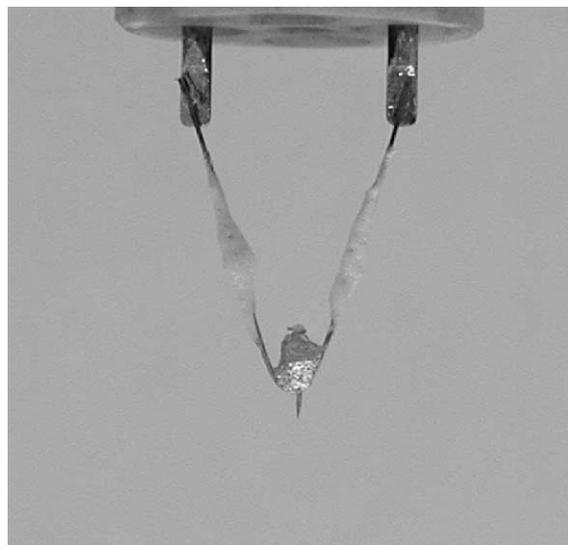


Fig. 2. Overall view of the filled Au–Dy–Si LMIS.

ExB filter. The output current was collected by a Faraday cup.

3. Results and discussion

3.1. Calculation of ternary alloy compositions for LMIS

Calculations of the Au–Si–Dy alloys were performed with the help of phase diagrams of binary alloys like Au–Si and Au–Dy, given in [9].

Suppose that a basic alloy consists of two completely miscible components *A* and *B*. In general, the most suitable binary alloys are those with a simple phase diagram and a single eutectic point. The change in Gibb’s free energy related to an equilibrium melting of a component *A* in a narrow compositional interval near the eutectic point may be written as

$$\Delta G_{A \text{ melting}} = 0 \quad \text{or}$$

$$RT \ln (X_{A \text{ liquid}}/X_{A \text{ solid}}) + \Delta H_{A \text{ melting}} - T \Delta S_{A \text{ melting}} = 0, \quad (1)$$

where $\Delta H_{A \text{ melting}}$ and $\Delta S_{A \text{ melting}}$ are the melting related changes in the sum of standard and excess molar enthalpy and entropy of component *A*, re-

94 spectively; $X_{A\text{solid}}$ and $X_{A\text{liquid}}$ are the molar frac- 131
 95 tions of the component A in the solid and liquid 132
 96 phase, respectively [10]. In the case of the Au–Si 133
 97 alloy 134

$$X_{\text{Au solid}} = X_{\text{Si solid}} = 1. \quad (2)$$

99 Similarly for the component B

$$\Delta G_{B\text{melting}} = 0 \quad \text{or} \\ RT \ln(X_{B\text{liquid}}/X_{B\text{solid}}) + \Delta H_{B\text{melting}} - T \Delta S_{B\text{melting}} = 0. \quad (3)$$

101 The eutectic condition is given by

$$\Delta G_{A\text{melting}} = 0 \quad \text{and} \quad \Delta G_{B\text{melting}} = 0. \quad (4)$$

103 Empirical values of the parameters $\Delta H_{A\text{melting}}$, 139
 104 $\Delta S_{A\text{melting}}$, $\Delta H_{B\text{melting}}$ and $\Delta S_{B\text{melting}}$ may be found 140
 105 on the basis of A – B binary phase diagrams using a 141
 106 set of related values of T , $X_{A\text{solid}}$, $X_{A\text{liquid}}$ and T , 142
 107 $X_{B\text{solid}}$, $X_{B\text{liquid}}$ near the eutectic point and mini- 143
 108 mizing the least square deviations of the values of 144
 109 $\Delta G_{A\text{melting}}$ and $\Delta G_{B\text{melting}}$ from zero. 145

110 Applying a similar procedure for the A – C bi- 146
 111 nary phase diagram, $\Delta H_{C\text{melting}}$ and $\Delta S_{C\text{melting}}$ may 147
 112 also be calculated. 148

113 The eutectic condition for three components is 149
 114 then defined by $\Delta G_{A\text{melting}} = 0$, $\Delta G_{B\text{melting}} = 0$ and 150
 115 $\Delta G_{C\text{melting}} = 0$. Taking into account that for any 151
 116 ternary alloy 152

$$X_{A\text{liquid}} + X_{B\text{liquid}} + X_{C\text{liquid}} = 1, \quad (5)$$

118 the eutectic values of $X_{A\text{liquid}}$, $X_{B\text{liquid}}$, $X_{C\text{liquid}}$ and 153
 119 the eutectic temperature (T_{eutectic}) may be calcu- 154
 120 lated. 155

121 In most cases, however, a composition of the 156
 122 ternary alloy with a fixed acceptable concentration 157
 123 of component C in the proximity of the eutectic 158
 124 point (i.e. $X_{C\text{liquid}} = \text{const}$) rather than the exactly
 125 ternary eutectic composition of the alloy is needed.
 126 In this case, $X_{C\text{liquid}} = \text{const}$ is pre-defined and the
 127 only conditions

$$\Delta G_{A\text{melting}} = 0, \\ \Delta G_{B\text{melting}} = 0 \quad \text{and} \\ X_{A\text{liquid}} + X_{B\text{liquid}} + X_{C\text{liquid}} = 1, \quad (6)$$

129 are satisfied when calculating the equilibrium 131
 130 $X_{A\text{liquid}}$, $X_{B\text{liquid}}$ and the melting temperature. 132

133 According to the previous considerations, an 134
 135 equilibrium composition of the Au–Si–Dy alloy 136
 137 containing 8 at.% Dy was calculated. This alloy 138
 139 composition contains 78.2 at.% of Au and 13.8 140
 141 at.% of Si and the calculated melting temperature 142
 143 is 294 °C. The vapor pressure of Dy is $<10^{-15}$ Pa at 144
 145 this temperature. 146

3.2. The Au–Si–Dy LMIS

147 The current–voltage characteristics of the Au– 148
 149 Si–Dy LMIS is shown in Fig. 3. This form is 149
 150 typical for such kind of sources. The curve has a 151
 152 hysteresis effect around the onset voltage. The 152
 153 threshold voltage is about 3.61 kV and the ex- 153
 154 tinction voltage about 3.56 kV. The reason is that 154
 155 a higher voltage is needed to form than to sustain 155
 156 the Taylor cone. A practically identical depen- 156
 157 dence is observed for an Au–Si–Pr LMIS [11]. It 157
 158 is worth to note that no significant changes in the 158
 159 electrical parameters of the Au–Si–Dy LMIS took 159
 160 place after 50 h of operation in the FIB system. 160
 161 According to preliminary data, the stability and 161
 162 time of operation of the Au–Si–Dy LMIS are 162
 163 similar to the widely used Au–Si–Be LMIS. 163

164 The mass spectrum of the Au–Dy–Si LMIS at a 164
 165 total ion current of 110 pA is presented in Fig. 4. 165
 166 The spectrum is characterized by the presence of 166
 167 lines attributed to emission of Si^{2+} , Si^+ , Dy^{3+} , 167
 168 Dy^{2+} , Au^{2+} , Au^+ , Au_3^+ and Au_2^+ . The ion current 168

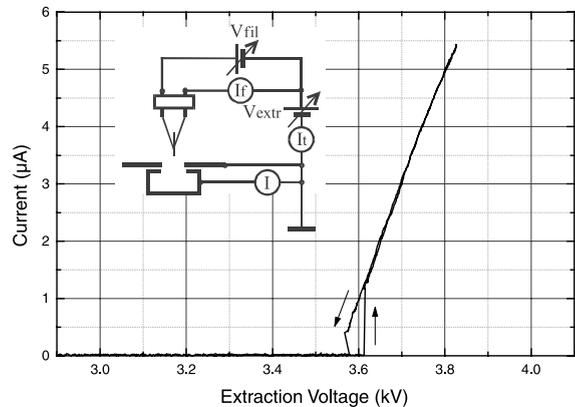


Fig. 3. Current–voltage characteristic of the Au–Dy–Si LMIS. The distance between the emitter tip and the extraction electrode is 2 mm. The heating current is 2.5 A.

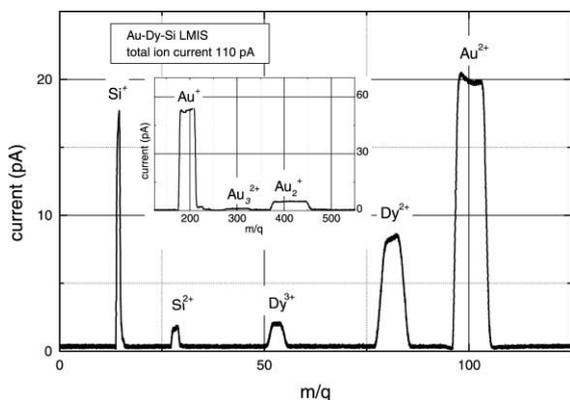


Fig. 4. Mass spectrum of the Au–Dy–Si LMIS at a total ion current of 110 pA and a total source current of 10 μ A.

159 is equal to about 1.6% of the total current for Dy^{3+}
 160 and 7.4% for Dy^{2+} , which makes the alloy prom-
 161 ising for implantation of Dy ions. It is interesting
 162 to note that we observe a substantial Dy^{3+} peak in
 163 the mass spectrum, although in an earlier investi-
 164 gation of a Dy–Ni LMIS this peak was not ob-
 165 served. This feature of Au–Si based alloys was
 166 found also for an Au–Si–Pr LMIS, where a larger
 167 percentage of Pr^{3+} presence was observed in
 168 comparison with the pure Pr LMIS [7]. The au-
 169 thors explained this difference by the influence of
 170 the Au and Si components on the physical prop-
 171 erties of the alloy and thus on the shape of the
 172 Taylor cone or on the field strength at the tip of
 173 the cone. Apparently, this is a common feature for
 174 any alloy and the ratio of differently charged ions
 175 in ion beams depends on the components of the
 176 selected alloy.

177 4. Summary

178 An Au–Dy–Si LMIS for Dy ion FIB implanta-
 179 tion was developed. The mass spectrum of the ion

beam shows the presence of high intensity Dy^{3+} 180
 and Dy^{2+} ions. The fabricated Au–Dy–Si LMIS 181
 has high stability and exhibits a long operation 182
 time. A simple technique of theoretical calcula- 183
 tions for eutectic composition of ternary alloys for 184
 LMIS was suggested on the basis of binary phase 185
 diagrams of the corresponding elements. 186

Acknowledgements 187

We gratefully acknowledge financial support of 188
 the DFG-SFB 491. 189

References 190

- [1] U. Doetsch, A.D. Wieck, Nucl. Instr. and Meth. B 139 191
(1998) 12. 192
- [2] A.D. Wieck, K. Ploog, Appl. Phys. Lett. 61 (1992) 1048. 193
- [3] P.D. Prewett, G.L. Mair, Focussed Ion Beams from Liquid 194
Metal Ion Sources, Research Studies Press Ltd., Taunton, 195
Somerset, England, 1991. 196
- [4] P.J. Wellmann, J.M. Garcia, J.-L. Feng, P.M. Petroff, 197
Appl. Phys. Lett. 71 (1997) 2532. 198
- [5] K. Ando, A. Chiba, H. Tanoue, Appl. Phys. Lett. 73 (1998) 199
387. 200
- [6] N. Theodoropoulou, A.F. Hebard, M.E. Overberg, C.R. 201
Abermathy, S.J. Pearton, S.N.G. Chu, R.G. Wilson, Appl. 202
Phys. Lett. 69 (2001) 3475. 203
- [7] F. Machalett, W. Wesch, R. Muehle, S. Barth, Rev. Sci. 204
Instrum. 69 (1998) 1336. 205
- [8] P.J.A. Sazio, S. Vijendran, W. Yu, H.E. Beere, G.A.C. 206
Jones, E.H. Linfield, D.A. Ritchie, J. Cryst. Growth 201– 207
202 (1999) 12. 208
- [9] Landolt-Boernstein, in: O. Madelung (Ed.), Group IV, 209
Phase Equilibria, Crystallographic and Thermodynamic 210
Data of Binary Alloys, Springer, Berlin, 1995. 211
- [10] A.D. Pelton, in: R.W. Cahn, P. Haasen, E.J. Kramer 212
(Eds.), Materials Science and Technology, Vol. 5, Phase 213
Transformations in Materials, P. Haasen (Vol. Ed.), VCH, 214
Weinheim, New York, Basel, Cambridge, 1991, p. 1. 215
- [11] F. Machalett, P. Seidel, R. Muehle, Rev. Sci. Instrum. 67 216
(1996) 1015. 217