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The ion beam sputtering facility at KURRI: coatings for advanced neutron optical devices

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Abstract

We describe a film coating facility for the development of multilayer mirrors for use in neutron optical devices that handle slow neutron beams. Recently, we succeeded in fabricating a large neutron supermirror with high reflectivity using an ion beam sputtering system (KUR-IBS), as well as all neutron supermirrors in two neutron guide tubes at BL06 at J-PARC/MLF. We also realized a large flexible self-standing m=5 NiC/Ti supermirror and very small d-spacing (d=1.65 nm) multilayer sheets. In this paper, we present an overview of the performance and utility of non-magnetic neutron multilayer mirrors fabricated with the KUR-IBS

1. Introduction

The slow neutron beam is a very powerful probe for material science, life science, and industrial applications. The demand for neutron beams in various research fields continues to increase, and large-scale neutron beam facilities based on proton accelerators, such as the J-PARC in Japan and SNS in USA, have been constructed; meanwhile, Europe and China have launched the ESS and CSNS, respectively. Small-scale neutron source development is also active [1]. Even at the large-scale facilities, the intensity and brilliance of the neutron source is still quite weak in comparison to X-rays from synchrotron radiation. As a result, high-performance neutron optical devices are very important, and the multilayer mirror is one of the most important devices for neutron beam handling. The multilayer mirror consists of alternating layers of two materials with different potential energies for the neutron. It has artificial lattice spacing (d-spacing) that produces one-dimensional optical potential for a neutron beam. Development of multilayer mirrors is still being widely conducted for application in bending, polarizing, shaping and focusing neutron beams. A supermirror is a stack of multilayer mirrors with gradually increasing d-spacing values. The supermirror is widely used as key device in the neutron guide tube [2]. To the best of our knowledge, the world's first full-scale

14 supermirror neutron guide tube was constructed in 1985 at the Kyoto University Research Reactor Institute (KURRI),
15 [3].

16

17 There are two kinds of PVD coating systems for fabrication of neutron optical devices at the KURRI. One is based on a
18 vacuum deposition technique [4], and the other is ion beam sputtering (IBS) [5]. A multilayer with small *d*-spacing and
19 supermirror with large-*m* are desirable for enlarging utilization efficiency in neutron scattering experiments. Here, *m* is
20 the maximum critical angle of the mirror in units of critical angle of natural nickel. The required number of layers is at
21 least proportional to the fourth power of *m*, according to Porod's law. The IBS technique enables us to fabricate smooth
22 layer structures with sharp edges. In addition, the adhesion force to the substrate with IBS deposition is much larger
23 than with vacuum deposition. In fact, the neutron optics group the Japan Atomic Energy Agency (JAEA) installed a
24 large-scale IBS system for neutron spectrometers at J-PARC/MLF [6-8], and succeeded in fabricating a large-scale
25 neutron supermirror with high reflectivity and low diffuse scattering even on a plano-elliptical glass substrate [9-10].
26 The effective substrate area at the JAEA-IBS system is 500 mm in diameter. It is possible to fabricate a lot of
27 supermirrors in a neutron guide tube and focusing mirror. On the other hand, the maximum substrate area at the
28 KUR-IBS was limited to 200 mm in diameter, and the effective substrate area was smaller than the designed value due
29 to the small ion gun size.

30 In general, DC magnetron sputter is popular for the production of neutron multilayer mirrors, since it is possible to
31 produce large sizes at reasonable cost with high throughput deposition [11-17]. The smaller size was particularly
32 limited in actual application. Recently, we succeeded in enlarging the effective deposition size without significantly
33 modifying the KUR-IBS. The throughput deposition rate was almost the same as that in our previous work [5, 18], and
34 production costs were also reasonable. In addition, we recently successfully fabricated all neutron supermirrors in two
35 neutron guide tubes using the KUR-IBS, the total length of which was 29 m at BL06 at J-PARC/MLF [19]. In this
36 study, we present an overview of the performance and utility of the non-magnetic neutron multilayer mirrors fabricated
37 by the KUR-IBS.

38 **2. Multilayer fabrication system and the performance of small-size multilayers**

39 Figure 1 shows the schematic structure of the KUR-IBS coating system, in which the outlet of the ion gun is 12 cm in
40 diameter. The system is a special modified version of the Veeco IBD-350. The ion gun is generally used to generate
41 Ar^+ ions. The accelerator voltage and current of Ar^+ ions can be controlled with the resolution of 1 V and 1 mA, and
42 typical values were from 120 to 240 mA, respectively. The deposition rate is proportional to the current, and it is
43 necessary to use high current for high throughput. However, a typical current was used with 120 or 150 mA, since the
44 smoothness of the layer and service life of the ion gun are better with low-current operation. There are two kinds of
45 vacuum pumps, the turbo-molecular pump and cryo-pump shown in Fig. 1. The outer size of the process chamber was
46 1.2 m wide x 0.9 m long x 1.1 m high. That doesn't include the ion gun system, vacuum pumps, stand, lord lock

47 chamber, etc.; the process chamber itself was thus not huge. It could reach up to 5×10^{-6} Pa vacuum degrees. In general

48 use, the base pressure before deposition was 5×10^{-5} Pa, and that during deposition was 1.3×10^{-2} Pa. It took half a day

49 to vacuum up to base pressure from atmospheric pressure. The deposition rate and quality of the layer also depended on

50 the tilt angle of the substrate holder, ϕ. In the case of a small multilayer, it was possible to deposit multilayers on five

51 consecutive wafers without breaking the vacuum of chamber using lord-lock system. The substrate was placed at the

52 rotation center of the substrate holder with a tilt angle ϕ of -65 degrees. It was possible for the substrate holder to move

53 with the tilt angle ϕ from 90 to -80 degrees, and to rotate up to 10 turns a min. Here, the tilt angle ϕ=0 corresponds to

54 the vertical direction, and the isolation valve between the process chamber and lord lock chamber could open at an

55 angle of 90 degrees. The angles of targets were fixed. It was possible to use 6 targets, and the switching time was less

56 than 20 s. The beam spot size at the target could be controlled. The typical deposition rate of NiC and Ti targets were

57 0.03 and 0.035 nm/s, and the each layer thickness was controlled by deposition time with constant accelerator voltage

58 and current. The minimum time resolution was 1 millisecond using an electric shutter.

59 Figure 2 shows reflectivity of a small d-spacing NiC/Ti multilayer mirror deposited on the same silicon wafer, and the

60 cross-sectional TEM image taken by Dr. Thomas Krist's group in HZB. In the TEM image, the black and white lines

61 indicate NiC and Ti layers, respectively. The reflectivity was measured with a MINE2 reflectometer at JRR-3 at JAEA.

62 The incident neutron wavelength was 0.88 nm, the resolution was 2.7% in FWHM, and the divergent angle was smaller

63 than 1 milliradian. The designed d-spacing and thickness distribution were 2.9 nm and 5%, respectively. The d-spacing

64 of 2.9 nm corresponds to the critical angle of an m=10 supermirror, and the distribution was determined in order to

65 reflect all cold neutron beams at the MINE2. The layer thickness was gradually changed by controlling the deposition

66 time, and the measured reflectivity was well reproduced by the theoretical line with the average d-spacing of 2.9 nm.

67 The theoretical line was calculated by a one-dimensional Schrödinger equation, including the effect of a surface

68 roughness of Debye-Waller factor of 0.55 nm. Here the nuclear potential values of NiC and Ti layers were ideal: 243

69 and -51 neV, respectively.

70 In order to fabricate a large-m supermirror and wide-band small d-spacing multilayer with high reflectivity, even with a

71 theoretically smooth surface and ideal layer structure, the required number of layers increases to the fourth power of m.

72 The surface and interface roughness of the multilayer increases with total thickness, and we tested the performance of a

73 stacking multilayer technique. It enabled us to realize very small d-spacing and wide-band monochromator, as shown in

74 Fig. 3(a). The d-spacing at maximum intensity was 2.89 nm, and the reflectivity was higher than 0.4 and was actually

75 used as a key device of a Doppler shifter for the production of ultra-cold neutrons [20]. The total number of layers for

76 the monochromator was 85060. The reflectivity was measured with the MINE2 reflectometer at JRR-3 at JAEA. The

77 monochromator consisted of four mirrors. Each was a thin silicon substrate, 0.3-mm thick, coated on both surfaces. In

78 other words, each mirror consisted of two multilayers and the thin silicon substrate. For the mirrors, the first and second

79 multilayers were deposited on the incident and backside of the silicon wafer to reflect the neutron beam. The numbers

80 of layers on the first and second multilayers were 10336 and 10929, respectively. Figure 3(b) shows the reflectivities of

3

81   the first and second mirrors, as well as that of a two-mirror stack. The figure clearly shows that both the reflectivity and

82   band-width were increased by stacking the different *d*-spacing multilayers.

83   Figure 4 shows measured reflectivity by an *m*=7 NiC/Ti supermirror, which consisted of two stacked mirrors. The layer

84   sequence was designed using an algorithm based on Pleshanov [21]. Each mirror also consisted of two multilayers. The

85   first multilayer was an *m*=5 NiC/Ti supermirror deposited on a thin silicon wafer 0.3-mm thick. The number of layers

86   was 4766. The layer thickness of the first multilayer (*m*=5 supermirror) was gradually increased from the silicon

87   surface. The second multilayer was a wide-band multilayer deposited on the backside of the thin silicon wafer. The

88   design *d*-spacing was from 5.82 to 4.76 nm, and the total number of layers was 7131. The layer thickness was gradually

89   decreased from the silicon surface. The reflectivity of the first mirror featured a big valley, around *m*=5.5, and

90   reflectivity loss was significantly improved by stacking the two mirrors. These results indicate that the stacking

91   technique is useful for a small number of special uses. It is not easy to achieve production of large amounts for using in

92   a neutron guide tube. It is more effective for use with very small *d*-spacing mirrors with wide bands; the influence of

93   the substrate is not negligible with an increase in the number of stacking mirrors and self-standing multilayers like

94   crystal is one of the ultimate goals.

95   **3.   Production of large multilayer neutron mirrors**

96   In section 2, we showed the high performance and utility of non-magnetic neutron multilayer mirrors fabricated with

97   the KUR-IBS. However, the maximum wafer size and thickness were limited to 200 and 3 mm, respectively. The small

98   size problem was really limiting the application. Size was limited by the load lock system and ion gun size.

99   Geometrically, the process vacuum chamber could accept insertion of discs up to 500 mm in diameter. We decided to

100   stop using the lord lock system and developed a new substrate holding system. The outlet of the ion gun size was about

101   12 cm in diameter, and the thickness of the uniform area was almost the same as the outlet size. Figure 5(a) and 5(b)

102   are schematic illustrations of the thickness dependence of the tilt angles ϕ= -65 and ϕ= -17 degrees, respectively. As

103   shown in Fig. 5(a), it was not necessary to rotate the substrate holder for small samples, while 5(b) shows that the

104   coating for a large sample was impossible without this rotation. The area of uniform thickness with ϕ= -17 was smaller

105   than that with ϕ= -65 degrees; however, the thickness dependence is similar to a triangle, as shown in Fig. 5(c).

106   In the cross-sectional geometrical view, it is possible to fabricate large multilayer mirrors by rotating the substrate

107   holder. When we introduced a collimator between the target and the substrate holder, we could get a larger uniform

108   layer; however, the throughput was dramatically reduced. We aimed to demonstrate that actual production of large-size

109   neutron mirrors is feasible even with the KUR-IBS, in which ion gun is not so large. In fact, large-size neutron

110   supermirrors were fabricated without any collimator by adjusting the deposition parameters. The layer thickness was

111   controlled by deposition time, and the current with constant voltage; the minimum time and current resolution were 10

112   s and 1mA, respectively. The time resolution was determined by the period of rotation of the substrate holder to obtain

113   uniform thickness in each layer. The new substrate holder moved with the tilt angle ϕ=-17 deg, and rotated 6 turns a

114   min. The typical deposition rates of the NiC and Ti targets were 0.02 and 0.03 nm/s, and the accelerator voltage and

115   current of the ion gun were 800 V and 150 mV, respectively. It took 1, 2, 3, and 5 days for fabrication of m=2, 2.5, 3,

116   and 4 NiC/Ti supermirrors, respectively, at one batch including vacuum pumping. Figure 6 shows reflectivities of the

117   m=4 NiC supermirror at a batch. As shown in the image insert in Fig. 6, four m=4 NiC/Ti supermirrors were deposited

118   on smooth glass 300 mm long x 70 mm wide. The reflectivities were measured with a SOFIA reflectometer at

119   J-PARC/MLF [22, 23]. The reflectivities at all points were high, although the critical angle of total reflection differed

120   little by position. Thus, we enlarged the effective deposition area to more than five times the previous condition and

121   successfully fabricated all neutron supermirrors in two neutron guide tubes of BL06 at J-PARC. These reflectivities

122   were measured and their performance was high [19].

123   **4.   Large flexible self-standing multilayer sheet**

124   In the previous section, we clearly demonstrated that a large-size multilayer mirror can be fabricated even with a small

125   ion gun. This technique enables us to fabricate supermirrors on curved surfaces for ellipsoidal focusing mirrors.

126   Recently, forming and polishing techniques for curved substrates have been dramatically improved [25, 26]; however,

127   it is still very difficult to develop a large-m and small d-spacing multilayer mirror because it takes a long time to

128   fabricate and the coatings can easily peel off from the substrate due to the increase of internal stress of the multilayer.

129   The stress increases with total thickness, and sometimes destroys the surface of the glass substrate. From the point of

130   view of simple material mechanics, the stiffness of the multilayer is proportional to the third power of the total

131   thickness. This implies that a large-m supermirror and small d-spacing multilayer mirror are feasible owing to the thick

132   total thickness. In fact, the total thickness of an m=5 NiC/Ti supermirror is larger than 10 μm, and it is easy handling

133   the shape compared to a small-m supermirror. As shown in Fig. 7(a) and (b), large-size self-standing m=5 NiC/Ti

134   supermirror sheets were peel off from the glass substrate without the breaking by controlling the adhesion force

135   between the glass surface and thickest NiC layer of supermirror. The concept of peeling technique is similar to

136   production of replica mirrors [27, 28]. The mirrors are copied from plane or cylindrical glass surfaces in the following

137   way: First, Ni monolayer or Ni/Ti multilayer is deposited on the glass substrates. Second, nickel or copper electroplated

138   on these films as a supporting foil in which thickness is thicker than 0.15 mm. This composite metal sheet can

139   subsequently be removed from the glass substrate. On the other hand, the self-standing sheet has no supporting foil and

140   consists of neutron multilayer mirror itself. As shown in Fig.7 (c), neutron flux can be increased by concentrically

141   nesting mirrors with the same focal length and curvature. It is one of ultimate goals for development of self-standing

142   large-m supermirror sheet. Here winding direction of the sheet is very important in order to approximate the required

143   shape. The incident neutrons were reflected by the inner side of the winding sheet and the inner and outer surface

144   should be the thickest NiC layer and thinnest d-spacing, respectively. In Fig.7 (b), the winding direction was controlled

145   in the right direction for the use of the nesting mirrors. We cut 5 cm squares from the outer circumference of the sheet

146   and affixed them on the glass plate with thin double-sided tape, 0.01 mm thick. As shown in Fig. 8, the reflectivity even

147   at the outer circumference was nearly the same as that of an ordinary flat supermirror on the glass substrate. It is

148   possible to put the layer on the inner surface of an ellipsoidal shape; however, it is still very difficult to control the

149   waviness, and it is necessary to improve the forming fixation for the use of axisymmetric focusing devices.

150 There are some applications for VCN experiments without the improvement, since the required form accuracy is not so

151 severe. In particular, this is a very promising device for the blade mirror of a Doppler shifter [20]. The incident angle to

152 the mirror is almost 90 degrees, and the waviness of the mirror is negligible. A smaller d-spacing wide-band mirror is

153 required in the use of an intense neutron wavelength. The incident neutron velocity corresponds to the rotating speed of

154 the mirror, and the weight of the mirror should be light. Fig. 9 shows the reflectivity of a self-standing very small

155 d-spacing multilayer in which the layers numbered 10000. A sharp Bragg peak can clearly be observed at $Q_z = 3.8 nm^{-1}$,

156 although the reflectivity (1.5×10^{-3}) was low. The low reflectivity is estimated with interfacial roughness of 0.7 nm.

157 The sputtering condition for large size was not optimized for the thinnest d-spacing. In the part cut off from the outer

158 circumference of the sheet, the effective deposition rate is high. Ni crystal was easy to grow and NiC layer was not

159 smooth layer. In fact, we observed relatively strong diffuse scattering. The surface roughness of the glass substrate was

160 not confirmed for the thinnest d-spacing. We are improving the reflectivity for practical use by optimizing sputtering

161 and substrate conditions for the thinnest d-spacing.

162

163 **5. Conclusion**

164 We described the ion beam sputtering system at Kyoto University Reactor (KUR-IBS) and the performance of

165 fabricated non-magnetic multilayer mirrors. In the case of initial maximum design of a deposition area smaller than 200

166 mm in diameter, the m=7 NiC/Ti supermirror and wide-band small d-spacing (d=2.9 nm) multilayer with high

167 reflectivity were realized by a stacking technique.

168 The effective deposition area was enlarged to 460 mm in diameter and high-performance large-size neutron multilayer

169 mirrors were fabricated, even though the size of the KUR-IBS ion gun was much smaller than the deposition area.

170 Large flexible self-standing sheets of m=5 NiC/Ti supermirror and very small d-spacing (d=1.65 nm) multilayer were

171 also realized. Although the application as a neutron-focusing device is still limited due to the waviness of the sheets,

172 the fabrication technique has been almost established and the throughput is high. The KUR-IBS is already more than 10

173 years old; however, it is still very useful and continues to be improved for the development of advanced neutron
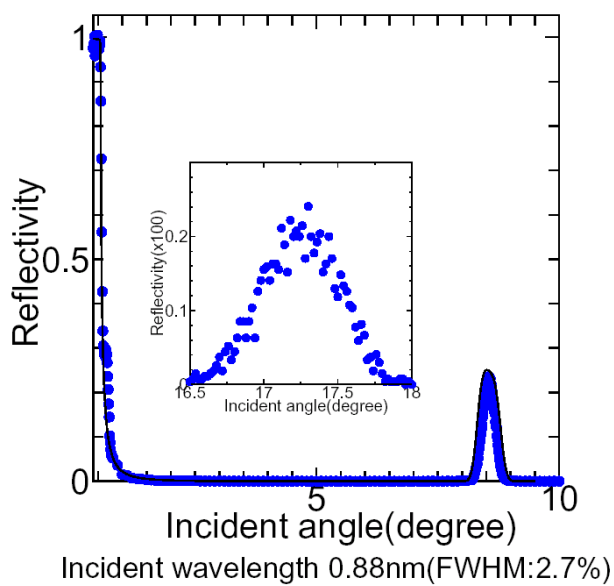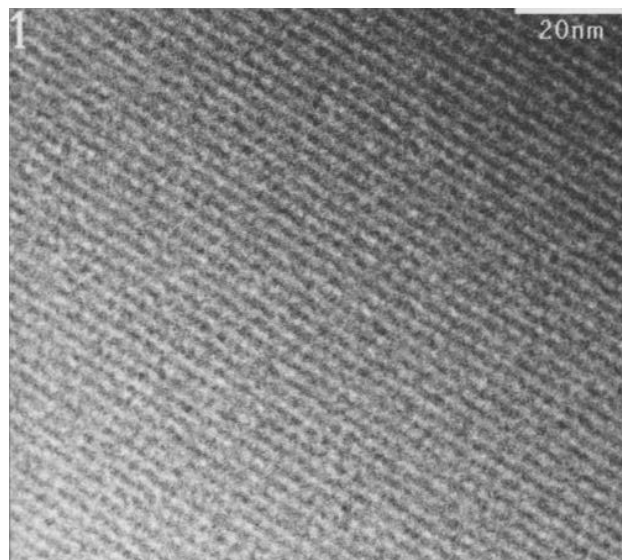
174 multilayer mirrors.

175

176
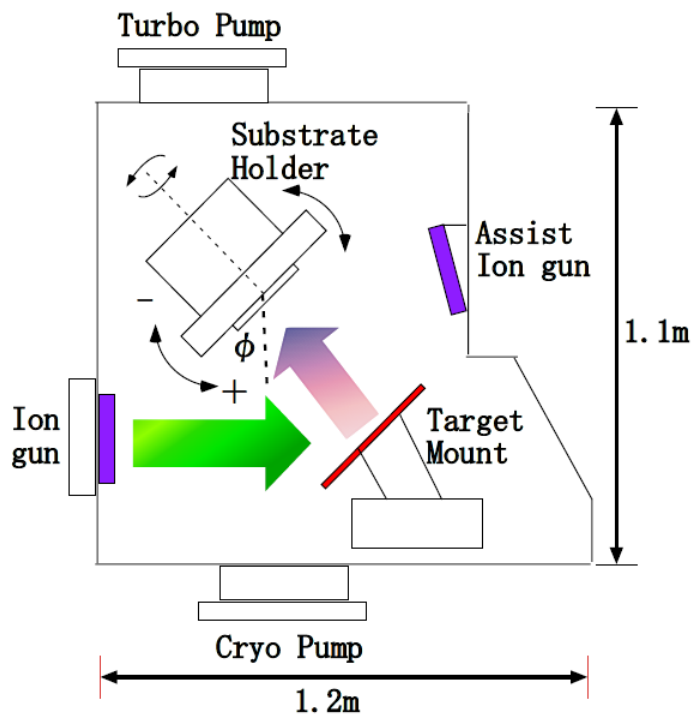
177

178   Fig. 1 Schematic view of ion beam sputtering system at KURRI.
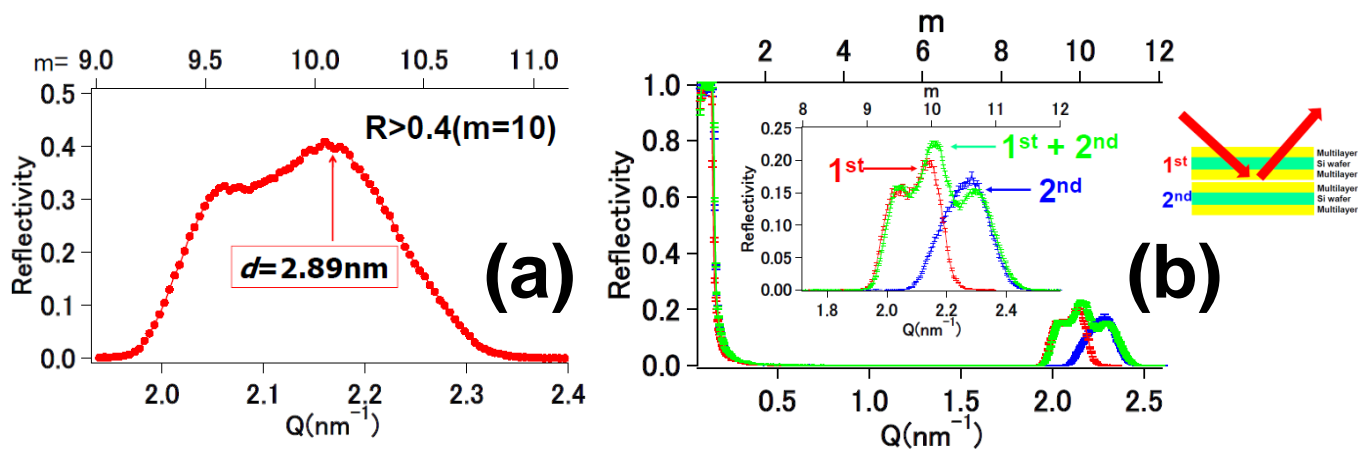
179

180

181   Fig. 2 Reflectivity of d=2.89 nm NiC/Ti multilayer mirror with 10000 layers; the cross-sectional TEM image. The
182   inserted graph shows reflectivity of the second order. The incident neutron wavelength was 0.88 nm.
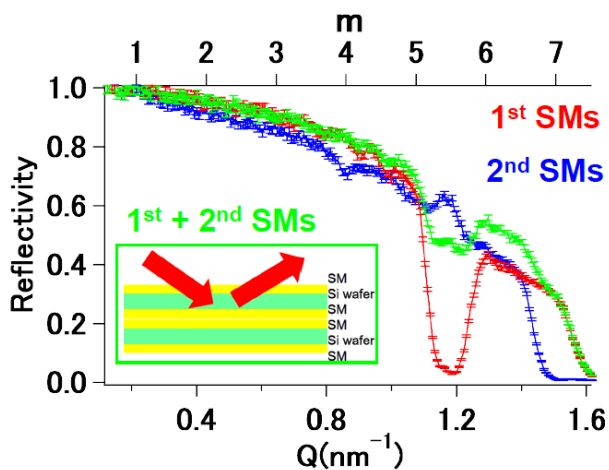
183

184

Fig. 3 Reflectivity of NiC/Ti stacking mirrors in with the following number of layers: (a) 85060; and (b) 10929(1st), 10336 (2nd), and 21265 (1st+2nd), respectively.

Fig. 4 Reflectivity of m=7 NiC/Ti supermirror stacking two mirrors, as shown in the insert.
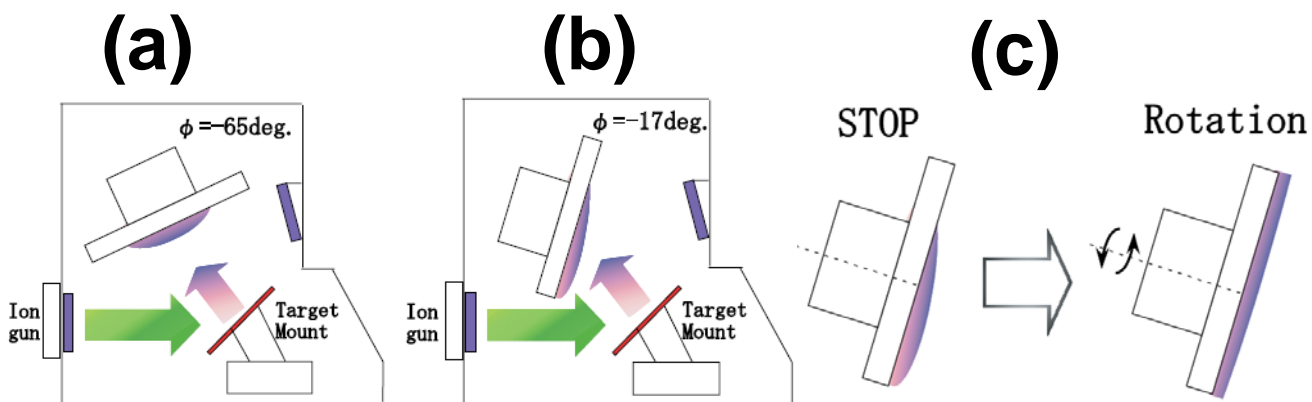
Fig. 5 Schematic illustration of thickness dependence on the tilt angle for (a) small, and (b) large samples in the case of stopping the substrate holder; (c) a schematic image of thickness dependence by rotating the substrate holder
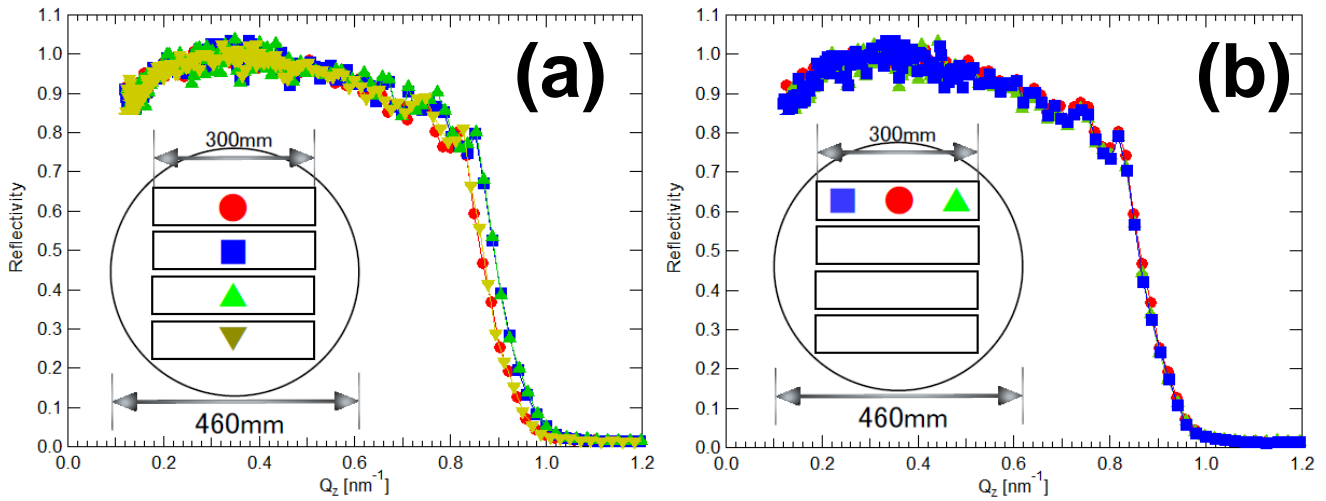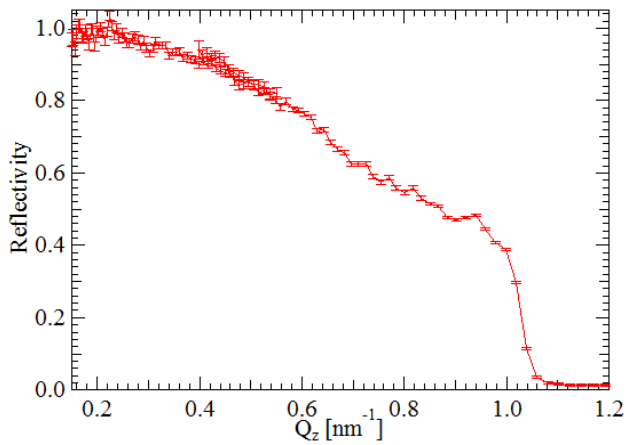
Fig. 6 Reflectivities of *m*=4 NiC/Ti supermirror with (a) the center of four mirrors, and (b) the left, center, and right of top mirror.
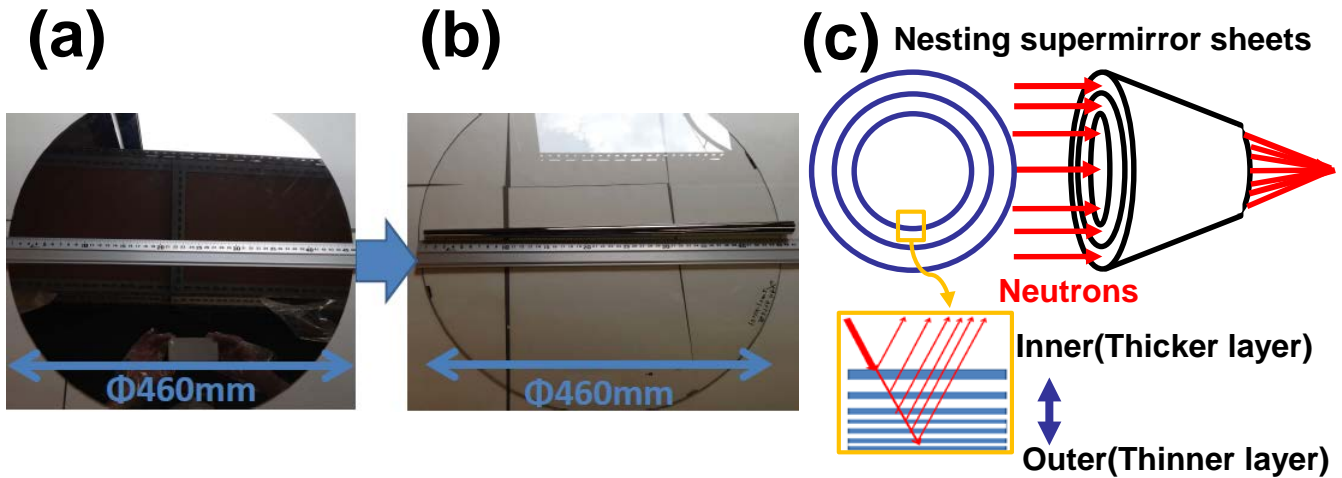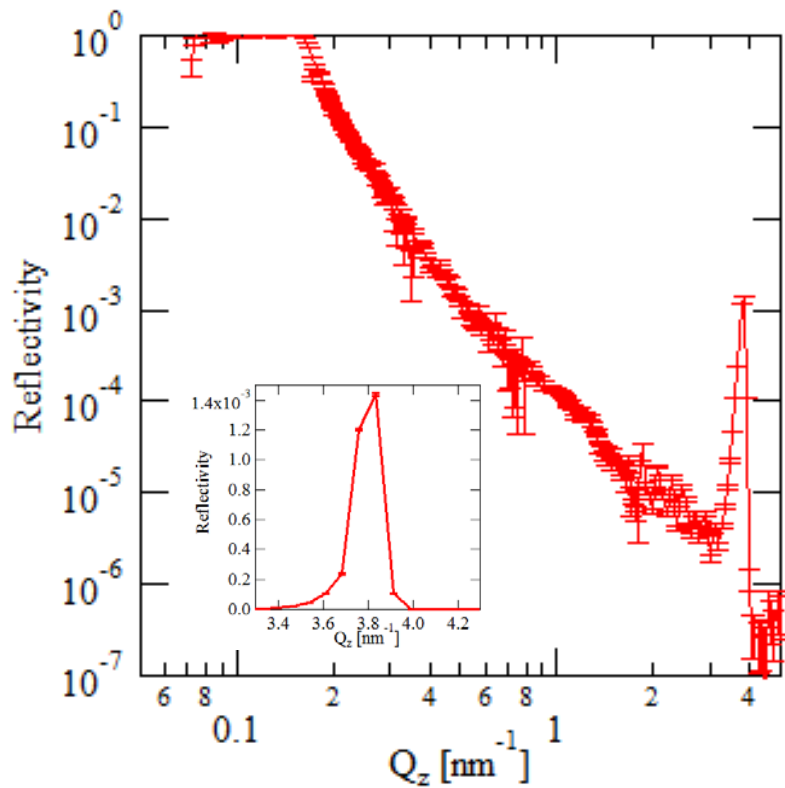

Fig. 7 Photograph of (a) *m*=5 NiC/Ti supermirror deposited on glass substrate and (b) the *m*=5 NiC/Ti self-standing supermirror peeling off from the glass substrate. (c) Schematic view of strong focusing device based on the nested axisymmetric supermirror sheets.

208

209 Fig. 8 Reflectivities of the *m*=5 NiC/Ti self-standing supermirror sheet affixed on glass plate.

210

211 Fig.9 Reflectivity of *d*=1.65nm NiC/Ti self-standing multilayer mirror in which layers numbered 10000. The insert
212 shows an enlarged view at 3.3<Q_z<4.3(nm).

213

Acknowledgments

222

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