NEW ROTATING ANODE X-RAY GENERATOR FOR XAFS EXPERIMENTS

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ABSTRACT

A high-power X-ray generator equipped with a lanthanum hexaboride cathode has been developed for X-ray absorption fine structure experiments. A high tube-current of more than 1,000 mA can be provided when operated at low tube-voltage of less than 20 kV. In addition, the focal width is narrow enough (less than 0.1 mm) to ensure good energy resolution. Extremely intense monochromatic X-rays (10^6 ~ 10^7 counts/(sec·mm^2) at the sample position), which are completely free from higher order harmonics and tungsten contamination lines, are available, when a Johansson-type spectrometer is employed. The filament life has been significantly prolonged by the high vacuum specification of the tube.

INTRODUCTION

X-ray absorption fine structure (XAFS) provides information on the atomic-scale structure around a specific atom, and has become an essential analytical tool in materials, biology and many other sciences.[1-3] While the availability of a synchrotron source has facilitated the application of XAFS,[2,3] instruments for carrying out measurements in an ordinary laboratory have been continuously developed and improved since the middle of 1970s.[4-6] Above all, a lot of work has been devoted to developing a spectrometer,[7-12] to produce monochromatic X-rays efficiently from a continuum spectrum generated in a tube. Further improvement has come with a special X-ray generator that is suitable for XAFS experiments,[13-15] since most conventional X-ray sources for industrial use have so far been designed for either diffraction or fluorescence experiments. The most powerful X-ray generator for XAFS[15] can realize 1,100 mA at 18 kV with a less than 0.1 mm line focus at 6 deg. take off, and therefore has capability of providing extremely intense continuum X-rays. However, the strong tungsten contamination lines from the filament material has still remained as a problem.

Table 1 Specifications of the X-ray generator for XAFS.

Anode	Molybdenum (10cm in diameter)
Filament	**Lanthanum hexaboride (LaB_6)** Promising material to obtain smooth spectral distribution without strong contamination lines
Allowable Load	20 kW Tube-Voltage 0 - 30 kV Tube-Current 0 - 1100 mA **Low tube-voltage operation** to suppress the generation of high energy X-rays. Capable of supplying extremely high intensity due to **extraordinarily high tube current.**
Focus Size	0.1 mm (width) x 10 mm (height) at 6 deg. take off **Narrow line focus** to obtain intense monochromatic X-rays without losing energy resolution
Vacuum	Around 1×10^{-7} Torr (when operating at full power) **High vacuum tube** to prolong life of filament

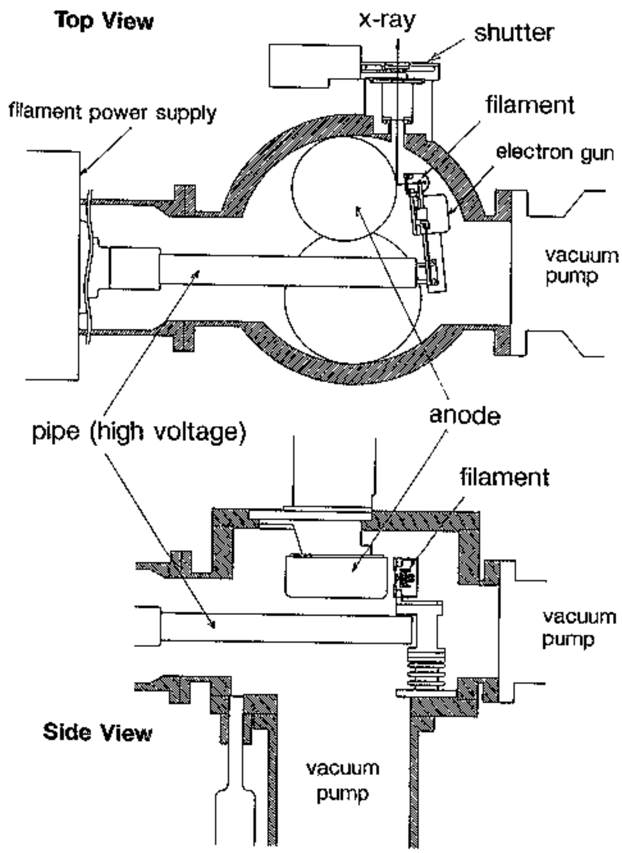

Figure 1 Structure of the X-ray generator for XAFS (top and side views)

Lanthanum hexaboride (LaB_6) is a promising electron emitter because of its small work function,[16] and is potentially a good alternative as a cathode material for an X-ray tube. However, it is not easy to get a good filament, because it is ceramic and therefore is difficult to be formed as a coil heater. Another problem is a short life, which depends on the temperature and the vacuum.[17] Though some valuable trials to employ a LaB_6 cathode in an X-ray generator have been reported,[18] the performance as a laboratory XAFS source (focal size, intensity at a sample position, low tube-voltage operation capability etc.) is still behind that of X-ray generator with a tungsten cathode. In the present paper, a new powerful X-ray generator for XAFS equipped with a LaB_6 cathode is described.

SPECIFICATIONS OF A NEW X-RAY GENERATOR

The problems of laboratory XAFS have been discussed by many authors,[3,5,6] and the main points are as follows: (1) the low intensity of incident X-ray flux at the sample position, resulting in insufficient count statistics of the signal, because XAFS uses monochromatic X-rays from a weak Bremsstrahlung component, (2) the degradation of the spectrum caused by 2nd or 3rd order harmonic reflections from a crystal monochromator, which is also significant at synchrotron sources[3] and (3) the effect of a non-smooth spectral distribution mainly due to the characteristic lines of tungsten, which is a conventional filament material and which evaporates onto the anode. One should note that all of them are due to the characteristics of the conventional X-ray source. The authors consider that the design of X-ray generator is important to solve these problems. As previously pointed out,[15] the essential specifications for an XAFS X-ray generator are as follows: (a) an extremely high tube-current (e.g. more than 1000 mA), (b) a narrow line focus (e.g. less than 0.1 mm at 6 deg. take off), (c) low tube-voltage operation (e.g. 10-30 kV), and (d) a filament free of emission lines which contaminate the useful part of the spectrum and/or has little evaporation (e.g. LaB_6).

The present X-ray generator employs a LaB_6 cathode with a molybdenum anode. The specification is listed in Table 1, and the schematic drawing is shown in Fig.1. The diameter of an anode is 100 mm, and it rotates at 6,000 rpm for cooling purposes by means of a motor installed inside the cup. LaB_6 material is commercially available (Cerac, Inc., Milwaukee, USA) and the resistivity data are found in the literature.[19] A filament is prepared by slicing a LaB_6 rod (1 inch diameter) and cutting with an electro-discharging wire. It works at much lower temperature (1750 ~ 2050 K) than a tungsten filament, but almost double the current was needed for heating because of its small resistivity. The size of the electron emitter plane is 11 mm × 1.3 mm, and the shape of the filament has been optimized to get uniform heat distribution and to avoid the effect of thermal expansion. The basic idea on the cathode structure for low tube-voltage operation is essentially the same as the previous study.[13] The tube is equipped with two turbo molecular pumps for improving the vacuum to prolong the life of a LaB_6 filament. In addition, the shape of the tube is carefully designed to this end. Its appearance is similar to that of a vacuum chamber for thin film process. The system pressure is around 1×10^{-7} Torr when operated at full power.

PERFORMANCES

The present X-ray source provides the maximum tube-current of 1,100 mA at low tube-voltage (14.5 ~ 18.0 kV) with a narrow line focus (0.1 mm × 10 mm at 6 deg. take off). When combined with a Johansson-type spectrometer,[14] intense monochromatic X-ray flux is available at the sample position. We observed 5×10^6 counts/(sec·mm^2) around Cu K edge (8.99 keV), when the generator was operated at 17.5 kV, 1,100 mA and Ge(220) (Rowland radius 350 mm, Johansson) was used as a monochromator with 1 deg. divergence and 0.1 mm receiving slits. Further increase in intensity beyond 10^7 counts/(sec·mm^2) is expected by evacuating X-ray paths and slightly widening slits. The 2nd order harmonic (~18 keV) was not observed because it is not generated in an X-ray tube. Such capability of controlling the spectral distribution of an X-ray source is an important feature which even a synchrotron does not posses. The measuring time for a sample of between 2 and 4 μt thickness is estimated to be usually around 30 min and at most 2 h for a difficult sample, when the accumulation count of transmitted X-rays and the point number of the spectrum are set as 10^6 and 300, respectively, and incident detector absorbs 20% of incident X-rays. Actually, we obtained the absorption spectrum of practical non-equilibrium materials in 40 min, and the results were in good agreement with synchrotron experiments.[20)]

This performance is of almost the same standard as our previous X-ray generator with a tungsten cathode,[15)] however, the strength is that the cathode consists of non-tungsten materials. Strong characteristic X-rays from tungsten evaporated and adsorbed on the anode affect the measurement of many important absorption edges between 7 and 12 keV. In our previous study, the tube-voltage had to be reduced below 10.2 kV so as not to generate tungsten L lines, resulting in an inevitable intensity loss. The present X-ray source equipped with a LaB_6 cathode gives a very smooth continuum spectrum in the energy region between 6.5 and 30 keV except the anode (molybdenum) characteristic lines. Since the evaporation rate of a LaB_6 filament is extremely low[18)], lanthanum emission lines (4 ~ 6.3 keV) in the spectrum were enough weak. We don't have to lower the power to avoid the contamination lines any more, and as a result, intensity at the sample position has been further improved. In the earlier stage of the development, the life of the filament was too short to use for practical measurement. However, through the improvement of the vacuum in the tube and designing the filament to optimize the operation temperature, the life has been prolonged to more than 2,000 h.

SUMMARY & ACKNOWLEDGMENTS

In conclusion, a new intense X-ray source for XAFS experiments was developed. It has a specially designed LaB_6 cathode and other components for providing a high tube-current at low tube-voltage with a narrow line focus. The maximum allowable current was 1,100 mA, and undesirable high energy X-rays and tungsten contamination lines were completely eliminated. Almost all of the problems in laboratory XAFS have been fixed by the present X-ray source. As for a transmission technique, it is now possible to measure a spectrum to the same degree of quality as that obtained from a synchrotron in a reasonably short time, typically 30 min ~ 2 h. The authors expect the present X-ray generator will play a role in extending the opportunities of XAFS study and further development of the X-ray absorption spectroscopy.

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