SPIDA : A Special Intense Diffractometer For Amorphous Materials

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A design concept is presented for a special intense neutron time-of-flight diffractometer for amorphous materials (SPIDA). This is a new-generation diffractometer for the study of the structure of amorphous and liquid systems requiring special environments and for a wide range of novel amorphous materials.

1. Introduction

It is now becoming clear that time-of-flight diffractometers for non-crystalline samples may be divided into two distinct and complementary types. The first type involves a very open design with a large number of detectors at low scattering angle 2θ. The designs of two such instruments, one at the ISIS source (SANDALS[1]) and one at the IPNS source (GLAD[2]), were reported at the ICANS-X conference in 1988, and both of these have now been constructed and are operating successfully. This type of diffractometer achieves a high count rate by the use of a large number of detectors, and the effects of inelasticity are minimised by the use of low values of 2θ. The study of the structure of non-crystalline samples of low atomic mass at ISIS has been significantly advanced by SANDALS since its start of operation in 1989. However, the necessary lack of secondary collimation (ie. shielding around the secondary flight path from sample to detector) in this first type of instrument has the consequence that whilst excellent results may be obtained for samples under ambient conditions, these instruments are not at all well suited to experiments which make use of extensive sample environment equipment due to the very high background signal always associated with an open design. Furthermore the lack of a large angular range is a severe disadvantage in the study of novel amorphous materials, firstly due to the lack of detectors with a high Q-resolution (where momentum transfer $Q=4\pi\sin\theta/\lambda$) and secondly due to the inability to perform anisotropy studies.

The second type of diffractometer involves a significant level of secondary collimation in the detector tank, and the detectors cover a very wide range of scattering angle. The LAD diffractometer[3], which has been essentially unchanged since it was constructed for use at the relatively low intensity Harwell Linac source[4], currently performs this role at ISIS. Experiments may be performed successfully on samples requiring a large amount of sample

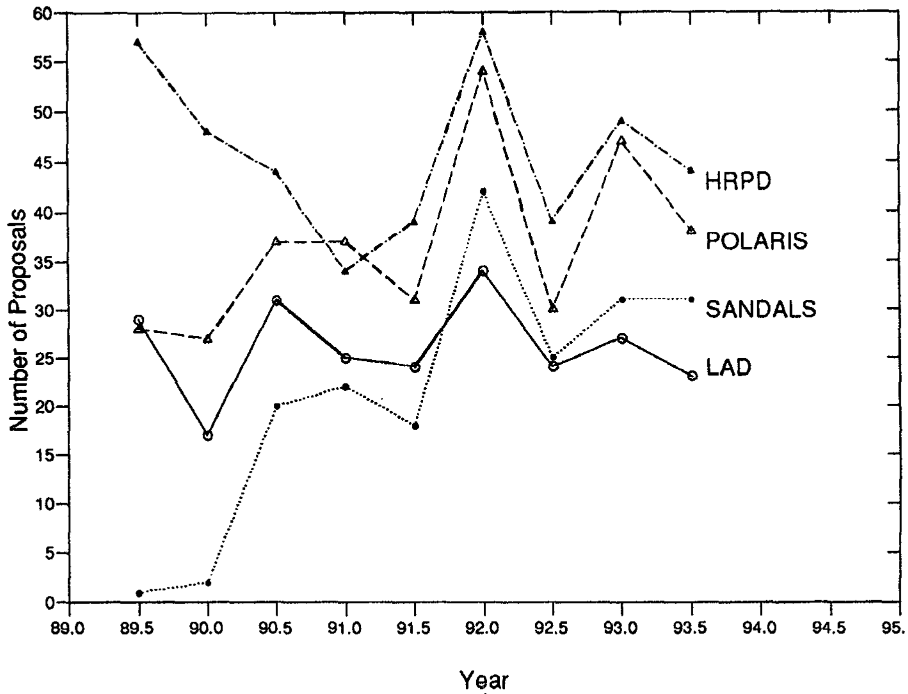

Figure 1. The number of ISIS diffractometer proposals since 1989.

environment equipment due to the high level of secondary collimation. The backward angle detectors in such an instrument have good Q-resolution, which can be of great importance in the characterisation of novel amorphous materials (in practice these may be crystalline to some extent), and also of use in the study of molten systems.

As an illustration of the demand for a non-crystalline diffractometer with good secondary collimation figure 1 shows the demand for diffractometer use at ISIS since the first receipt of proposals to use SANDALS. The steady demand for the present LAD diffractometer has been unaffected by the arrival of SANDALS, demonstrating that the two types of non-crystalline diffractometer are truly complementary and that there is a clear scientific need for diffractometers of the second type discussed above.

2. Design Criteria

For a diffractometer of the type discussed here the foremost design criterion concerns the secondary collimation; this must be designed so that each detector element views the whole of the illuminated volume of the sample, but as little of the surrounding sample environment equipment as possible so as to minimise background counts. The detector arrangement should then be designed so as to achieve the maximum intensity and the maximum range in both Q and 2θ without compromising the secondary collimation requirements.

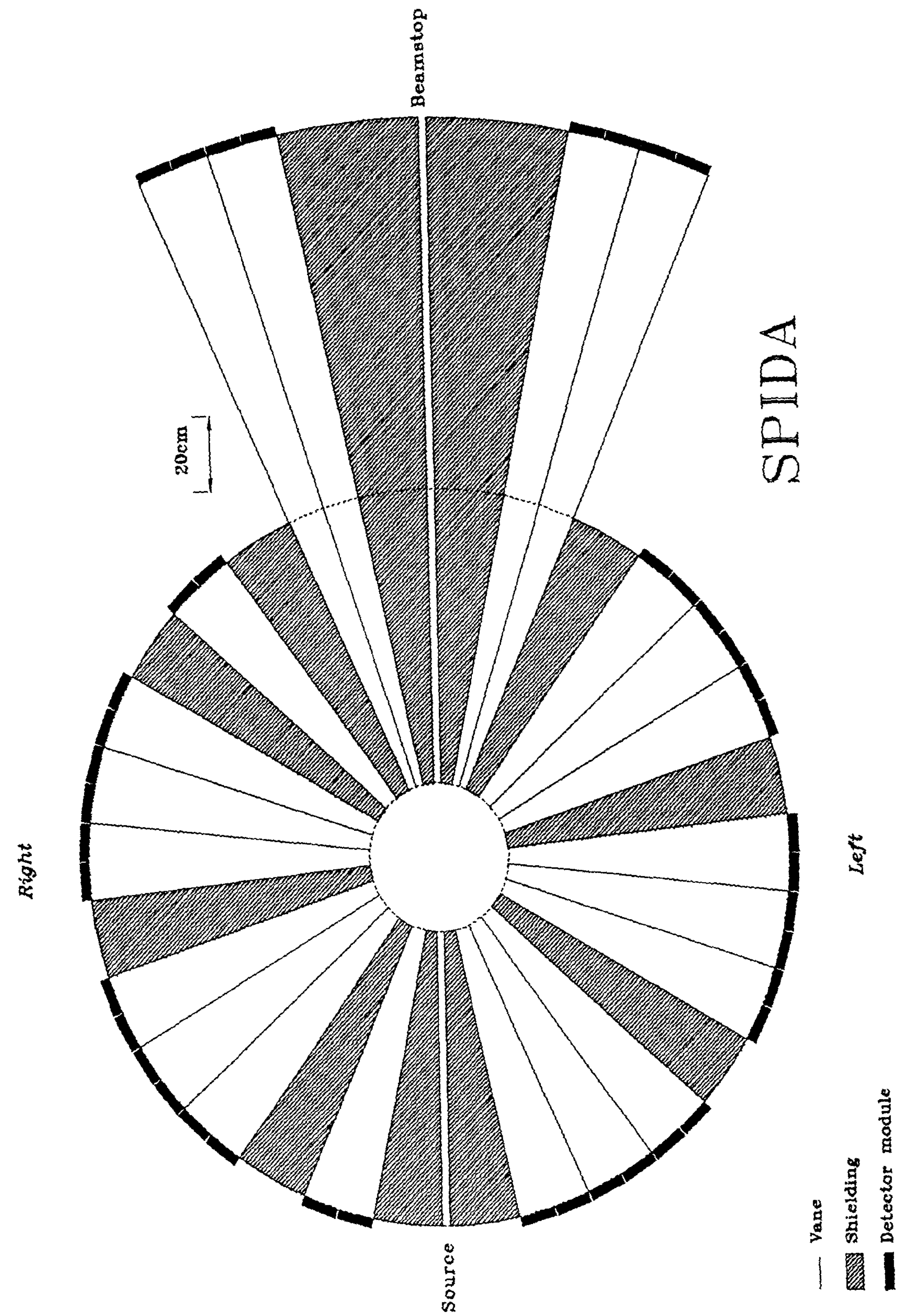

Figure 2. A plan view of the SPIDA instrument.

3. Specification

The SPIDA instrument, as shown in figure 2 and specified in tables 1 and 2, has been designed to meet the scientific requirements described above in section 1 and the design criteria given in section 2.

Moderator:	methane 110K
Incident flight path:	10m
Scattered flight path:	1m
Scattered flight path (low angle):	2m
Beam size:	rectangular 15mm wide and 40mm high
Beam:	S7
Detectors:	ZnS scintillator modules 10cm wide and 30cm high
Sample tank:	standard ISIS 'Tomkinson hole'

Table 1. Some design specifications for SPIDA

Howells[5] has shown that a cooled moderator is to be preferred for non-crystalline time-of-flight diffraction since this gives rise to inelasticity corrections which are better behaved than for an ambient moderator. Hence SPIDA is designed for use on the 110K liquid methane moderator at ISIS.

At all but the lowest and the highest scattering angles the SPIDA design involves having one detector module situated in the horizontal plane with two more situated above and below. In this way the detector solid angle, and hence the count rate, is maximised.

Theoretically the minimum Q which can be achieved by the low angle detector bank is 0.19Å^{-1}. However, in practice the lowest reliable Q is determined not by this theoretical limit but by the point at which the data become unreliable due to strongly rising background contributions at low Q. For this reason it is necessary for the low angle detector banks to have a very low intrinsic background level. The secondary flight path of the low angle detectors is longer than that of the other detectors firstly to facilitate a high level of secondary collimation to reduce backgrounds and secondly to improve resolution.

right	*left*
12°-24° (2m)	12°-24° (2m)
36°-48°	36°-72°
60°-96°	84°-120°
108°-144°	132°-168°
156°-168°	

Table 2. The range of scattering angle 2θ covered by the detector banks

The detector arrangement is asymmetric in order to achieve a wide and continuous angular coverage, without compromising the secondary collimation requirements. A notable application of this arrangement is in the study of anisotropy in planar samples in which case a wide range of relative orientations between scattering vector **Q** and sample can be achieved simultaneously[6].

Soper[1,7] has described how the intensity of diffractometers may be compared by use of the C-number, defined to be the count rate per inverse Ångstrom per unit volume of standard scatterer (usually vanadium);

C-number = neutrons /second /$Å^{-1}$ /cm^3 of vanadium (1)

Figure 3 shows the measured C-number of the LAD diffractometer together with calculations of the C-number for a completed SANDALS and for the SPIDA design, based on data from the present SANDALS. For comparison note that the D4B diffractometer at the ILL reactor has C-number=53 n/sec/$Å^{-1}$/cm^3V at a wavelength of 0.7Å. Clearly the SPIDA design presented here is capable of a very good count rate, compatible with its secondary collimation requirements.

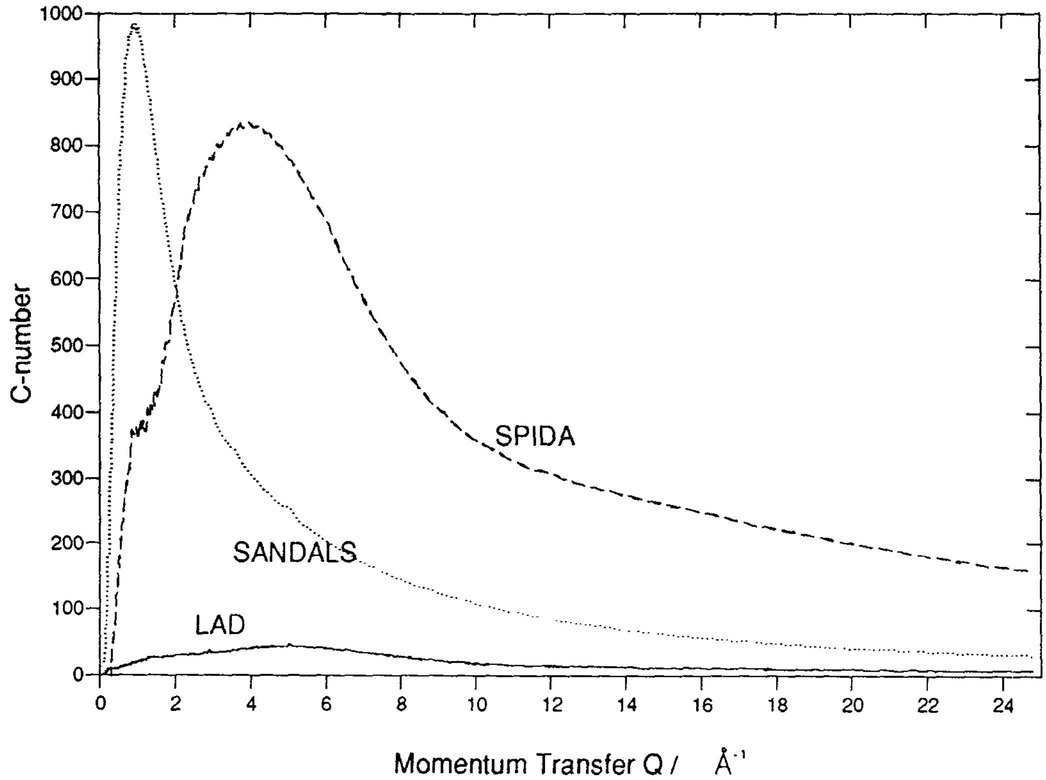

Figure 3. The C-number for SPIDA (dashed line), for a completed SANDALS (dotted line) and for LAD (continuous line).

4. Conclusions

A design concept for a new-generation non-crystalline time-of-flight diffractometer with good secondary collimation has been presented and shown to be able to produce a high count rate. Such an instrument is well-suited to the study of novel amorphous materials and liquid systems in high background sample environment conditions which continue to be of major scientific importance, and also to anisotropy studies on oriented samples.

References

[1] A.K.Soper, in 'Advanced Neutron Sources 1988' ed. D.K.Hyer, IOP Conf. Series **97**(1989)353.
[2] R.K.Crawford, D.L.Price, J.R.Haumann, R.Kleb, D.G.Montague, J.M.Carpenter, S.Susman and R.J.Dejus, in 'Advanced Neutron Sources 1988' ed. D.K.Hyer, IOP Conf. Series **97**(1989)427.
[3] W.S.Howells, 'A Diffractometer for Liquid and Amorphous Materials at the SNS', RAL Report RAL-80-017 (1980).
[4] C.G.Windsor and R.N.Sinclair, Phys. Bull. **33**(1982)290.
[5] W.S.Howells, Nucl. Instr. and Meth. **223**(1984)141.
[6] G.R.Mitchell, F.J.Davis, R.Cywinski and A.C.Hannon, Polymer Commun. **30**(1989)98.
[7] A.K.Soper, W.S.Howells and A.C.Hannon, 'ATLAS - Analysis of Time-of-Flight Diffraction Data from Liquid and Amorphous Samples', 1989, Rutherford Appleton Laboratory Report, RAL-89-046.