1.1 Preliminary SANS Studies on Nanocrystalline and Polymer Systems

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ABSTRACT

We reported some preliminary data on SANS experiment from Nd-Fe-B nanocrystalline commercial sample. The profile at even 12 m SDD still could not include the peak. Some polymer systems representing graft (ABS) and blend (ABS-LDPE and ABS-PP) polymers are also investigated. The profiles are not very satisfactory.

INTRODUCTION

The existence of BATAN's SANS spectrometer has been socialized and many prospective users are eager to take advantage. Before we can run their samples, we'd like to make sure that what will be the results of the SANS experiments are real and true. In the last few months, we were struggling to take some SANS data from a nanocrystalline and polymer systems. This short paper will present some of the results and we'd like to have suggestions in pursuing these activities.

NANOCRYSTALLINE SAMPLE

It is an Nd-Fe-B system which was bought commercially. Apart of what we have known about its magnetism, we only know that this sample has grain size of about 35 nm. The interest for this sample is to confirm that this grain size is true and we'd like to show that BATAN's SANS spectrometer can do that.

Figure 1 shows the SANS profiles for this sample taken at different sample-detector distances. It seems that the peak (at least part of it) is still behind the beam stopper. Eventhough this sample has a very high scattering length in the small angle area, we were not sure to run it at longer sample-detector distance (SDD). This is also limited by the low intensities for water at this very long SDD. Besides that, we still couldn't eliminate the parasitic signal when we go for long SDD, especially with fourth collimator in use.

We also took the diffraction pattern at bigger scattering angle using HRPD. Figure 2 shows the diffraction patterns taken at different temperatures. It seems that there is no change in the structure, what ever it is.

POLYMER SYSTEMS

The polymer group in our institute wants to utilize the SANS spectrometer for their activities. We have shown its capabilities for investigating block copolymer (SPU samples) and core-shell (SDS samples) systems in the last sub-workshop in Serpong. We knew that the results were not very satisfying, but at least we have done such things and hopefully could improve the results later.

Lately, we were trying to see the possibility of investigating graft and blend polymers. These topics are also listed in the activities of collaboration set in Yogyakarta in the last workshop for SANS polymer project. We were trying to see the SANS profile from ABS (acrylonitryl-styrene-butadiene) as well as its mixture with various percentages of LDPE (low density poly-ethylene) and PP (poly-propylene).

The ABS which has soft and hard segment (like SPU) gave SANS profile as presented at the top of figure 3. It seems that the sample has a structure producing a peak at a certain q value, but the peak is still behind the beam stopper. Figure at the bottom part is a SANS profile of sample containing of 50%ABS - 50%LDPE. We can observe the changing of the profile compared to the all ABS content. These profiles were taken at 3m SDD. In order to see all the peak completely, we move the detector to 6m position.

Figure 4 shows a SANS profile from the sample containing 100% ABS and taken at 6m SDD with non-reflecting tube in place. We notice that there is an extra peak at left of the profile which we always regarded as parasitic signal. This always happens when we remove the guide tube and replace it with non-reflecting tube. SEM pictures taken from different compositions of ABS-LDPE mixture is given in figure 5.

The effect of 50%PP in the ABS sample is presented in figure 6. The 100% PP sample gave SANS profile as shown at the bottom of figure 6. Since both of the profiles were taken using non-reflecting tube, we could see the parasitic signal at the left hand side. Table 1 shows some of the mechanical and physical properties of those polymer samples that have been obtained using proper techniques.

SUMMARY

We have taken some SANS profiles from samples containing nanocrystalline and polymer systems. The results were not satisfactory and it seems very difficult to analyze. We'd like to have some suggestions. If possible, some beam time for those samples so we can compare the results and find the root of the problem. This will be very beneficial to BATAN's SANS spectrometer and its utilization in the future.

TABLE 1. MECHANICAL AND PHYSICAL PROPERTIES OF SOME BLEND POLYMERS

POLYBLEND ABS-LDPE

No.	Composition	Yield (kg/cm^2)	Tensile Strength (kg/cm^2)	Elongation at break (%)	Hardness (Shore A)
1	100 % ABS	208,53	210,14	178,57	97,0
2	5 % LDPE	184,47	---	207,14	97,0
3	15 % LDPE	157,57	---	2,86	96,3
4	25 % LDPE	140,29	---	---	97,3
5	40 % LDPE	86,57	---	---	97,0
6	50 % LDPE	73,89	---	---	96,3
7	60 % LDPE	68,71	---	---	95,0
8	70 % LDPE	65,92	210,83	5,71	95,0
9	100 % LDPE	106,34	126,21	522,86	95,0

POLYBLEND ABS-PP

No.	Composition	Yield (kg/cm^2)	Tensile Strength (kg/cm^2)	Elongation at break (%)	Hardness (Shore A)
1	100 % ABS	208,53	210,14	178,57	97,0
2	5 % PP	205,88	205,37	195,71	
3	15 % PP	152,33	---	---	
4	25 % PP	124,19	---	---	
5	40 % PP	116,78	---	---	
6	50 % PP	110,20	---	---	
7	60 % PP	139,65	---	---	
8	70 % PP	158,74	---	---	
9	100 % PP	322,88	429,57	828,57	

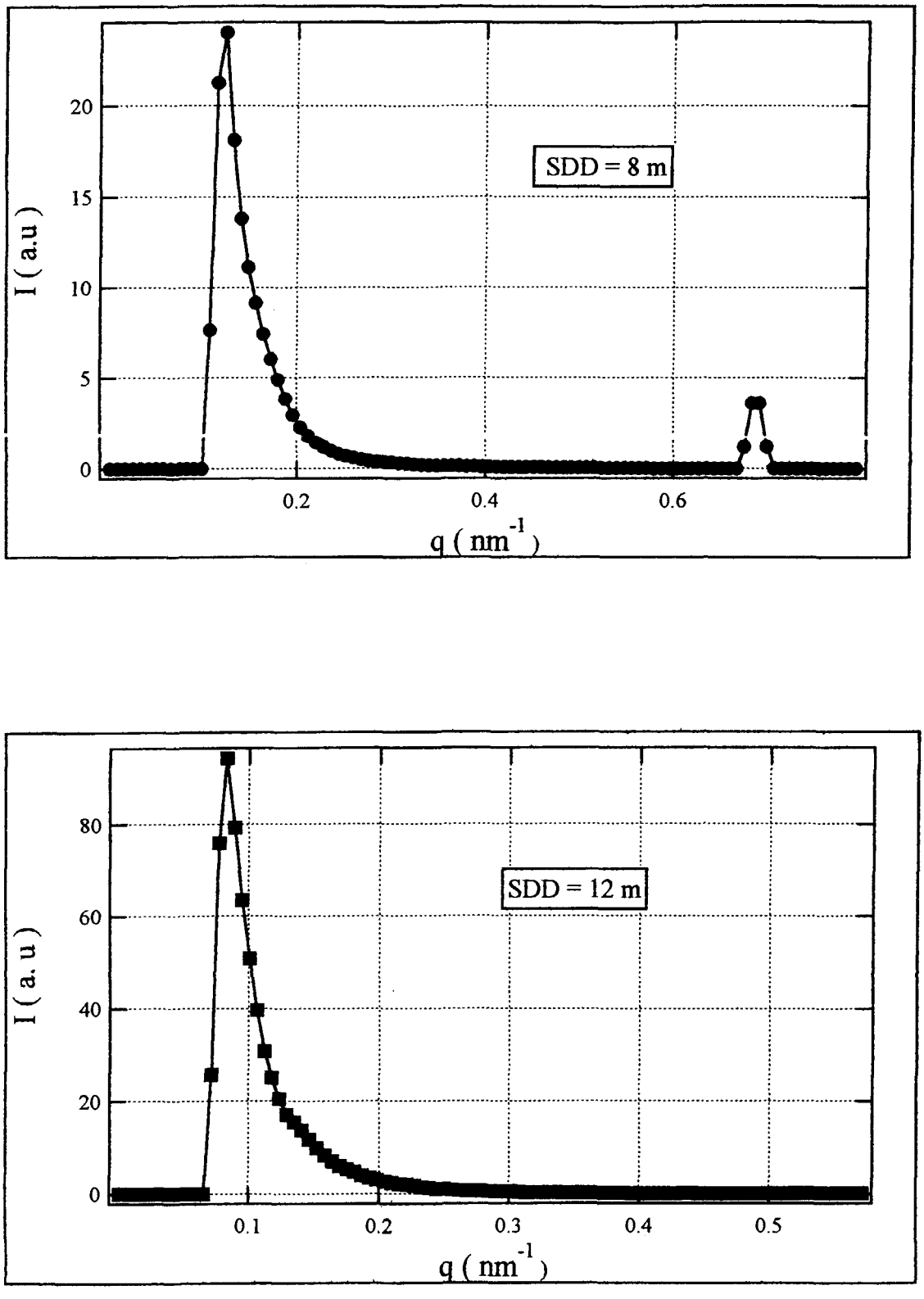


Figure 1. SANS profiles of Nd-Fe-B sample taken at SDD of 8 m and 12 m

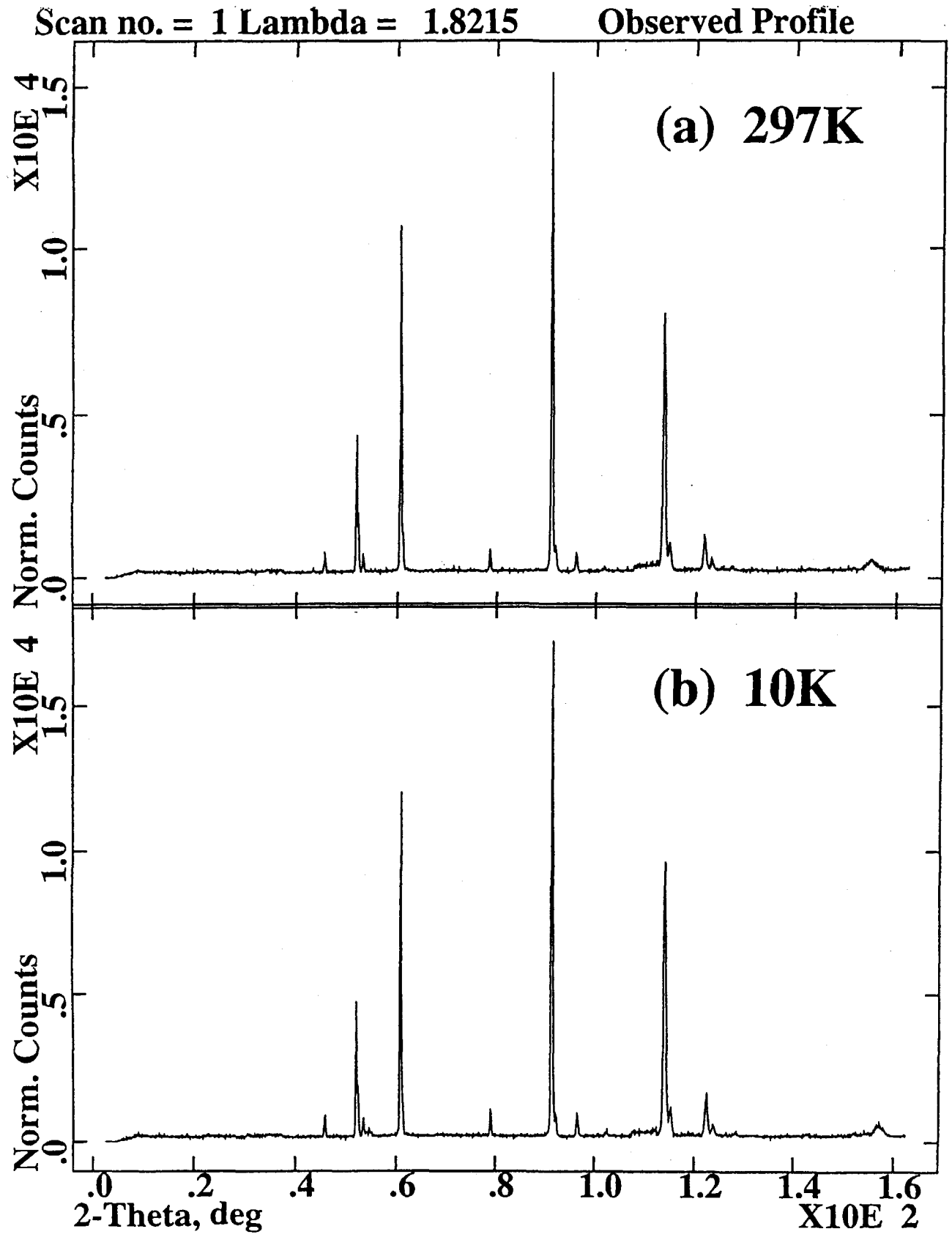


Fig 2. Wide angle diffraction pattern of Nd-Fe-B

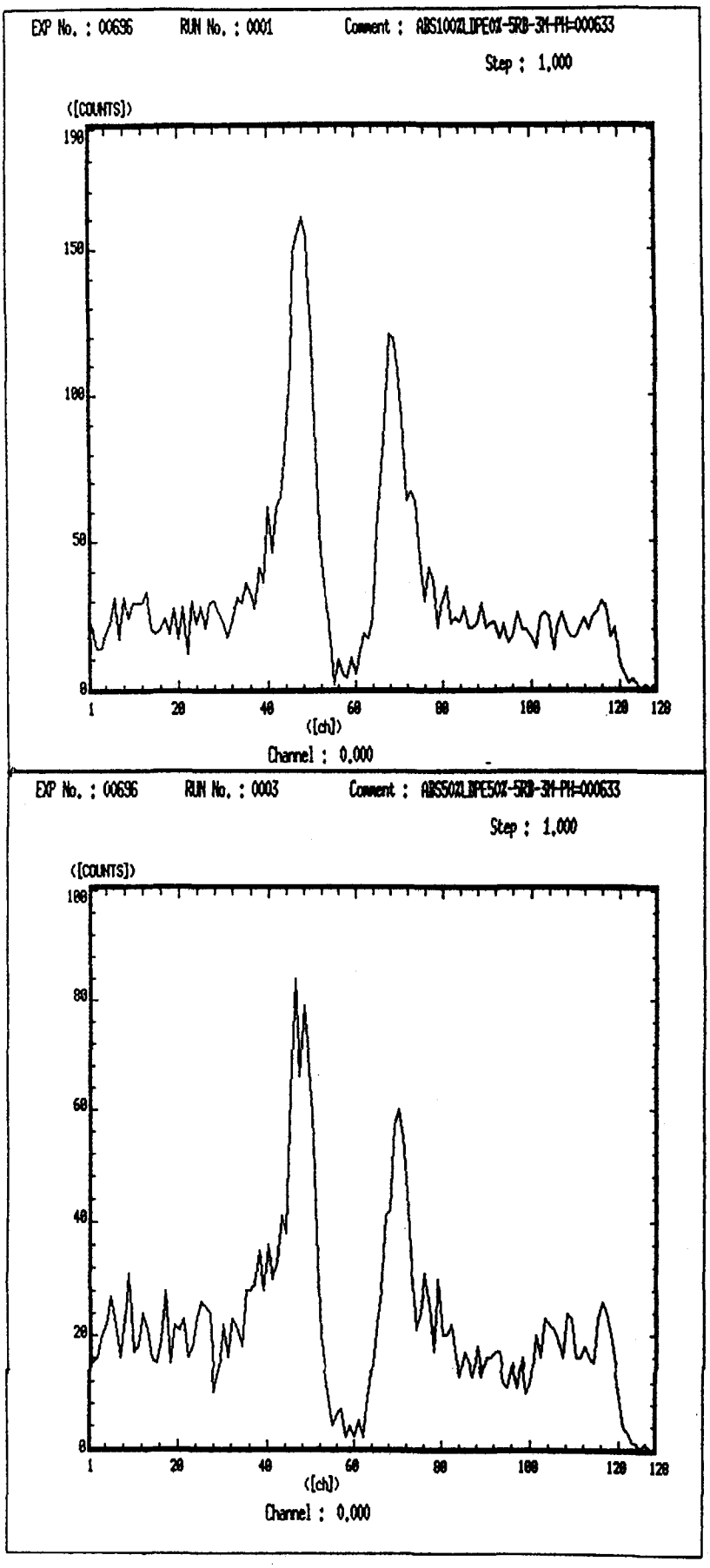
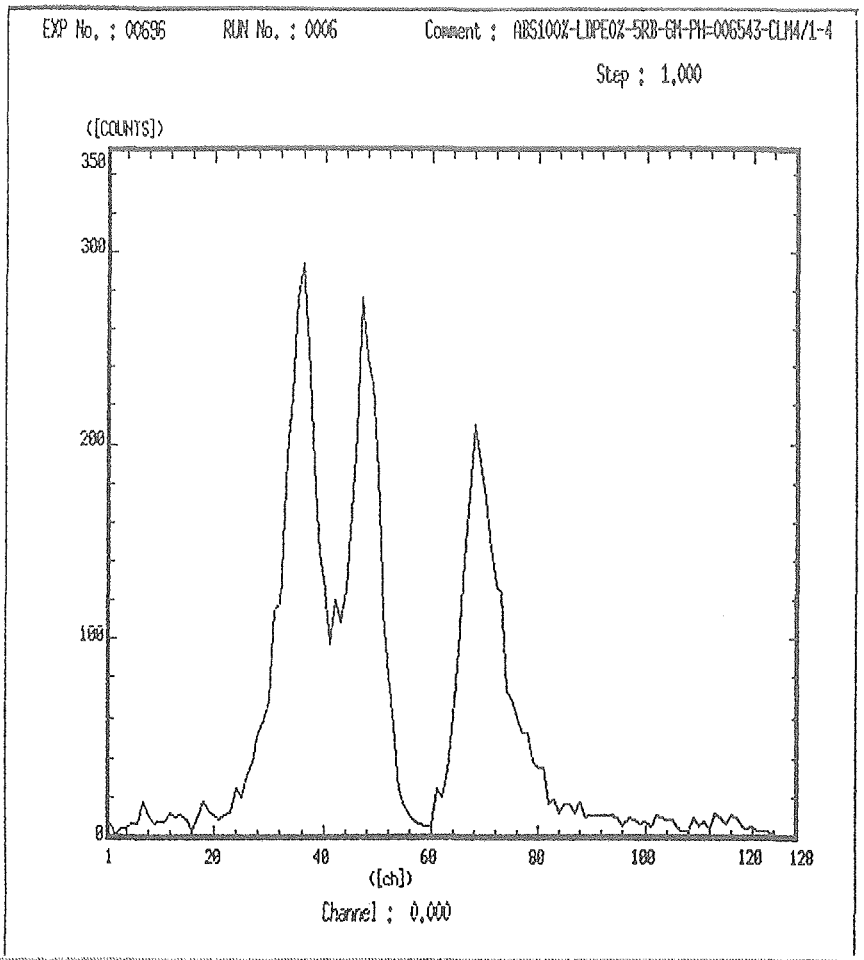


Figure 3 SANS profiles of 100% ABS (TOP) and 50% ABS - 50% LOPE (BOTTOM)



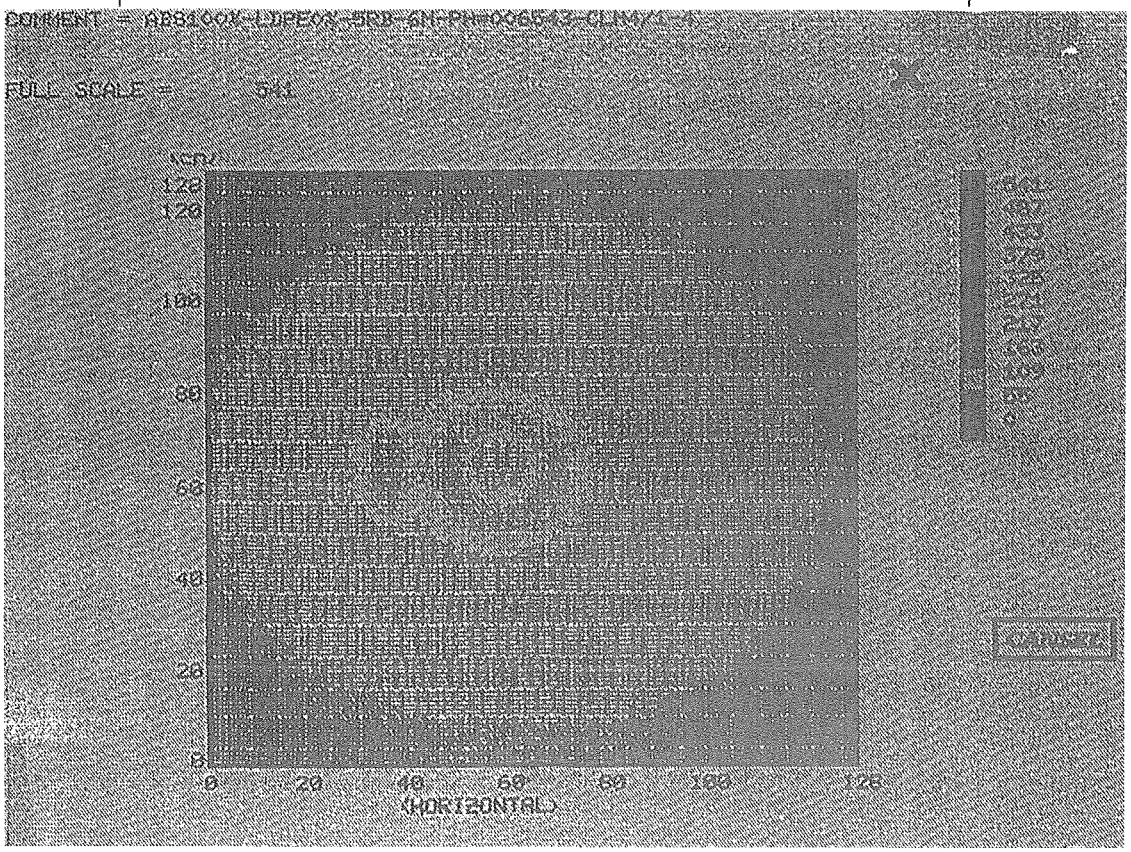


Figure 4 SANS profile of 100% ABS at 6m SDD

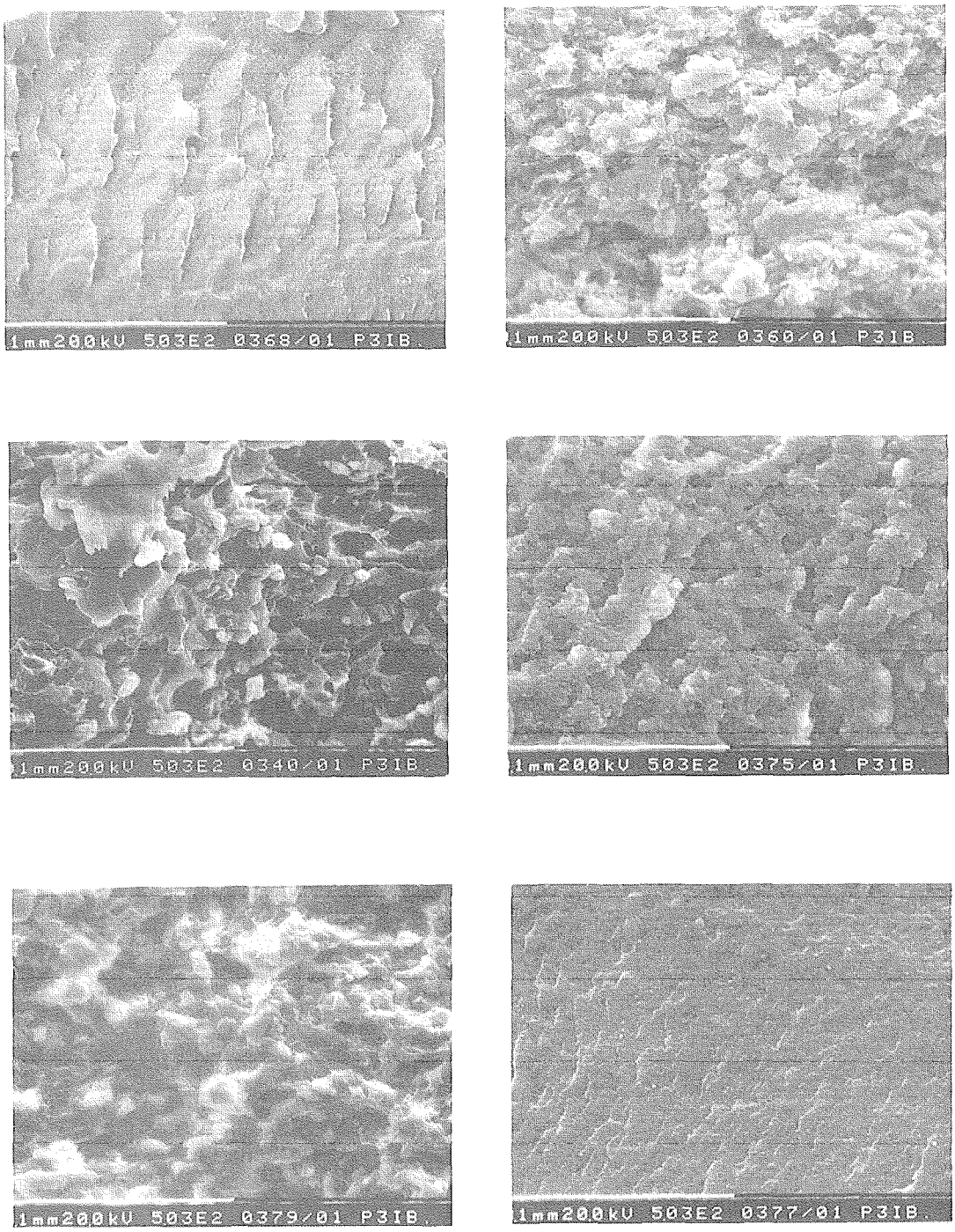


Fig.5. SEM Pictures of ABS-LDPE Blend Polymer with Different Composition

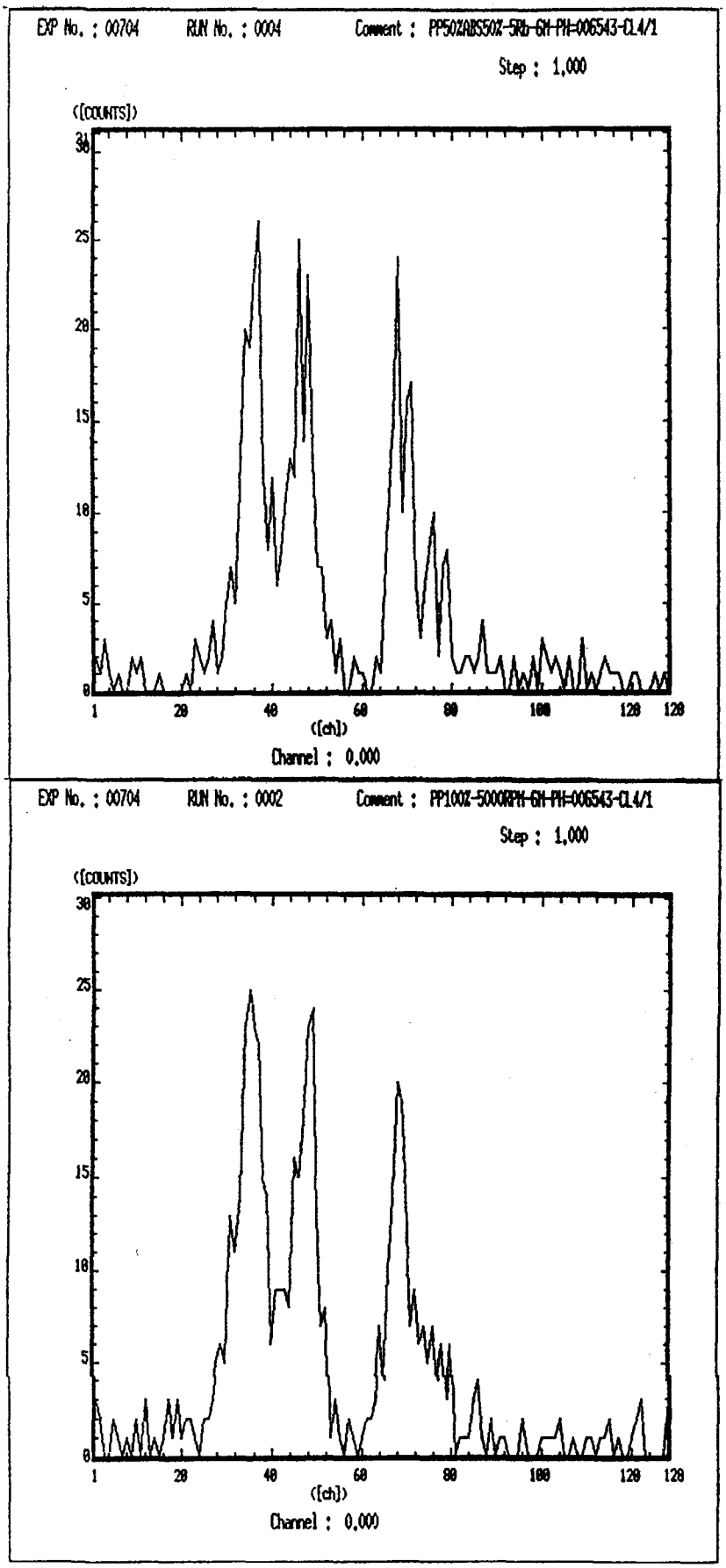


Figure 6 SANS profiles of 50% ABS - 50% PP(TOP) and 100% PP(BOTTOM)