

Using the anisotropy of magnetic susceptibility to infer flow-induced orientation of anisotropic particles: feasibility and sensitivity

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Abstract In this paper, the use of anisotropy of magnetic susceptibility (AMS) measurements has been investigated in order to check the sensitivity of this technique versus the flow conditions. The orientation of anisotropic magnetic particles during the flow of a polystyrene/magnetite blend through a capillary rheometer has been studied. Thanks to the magnetic properties of the magnetite, AMS measurements are possible. Different values of the filler concentration, viscosity, and shear rate were used. It is shown that the AMS technique is able to detect accurately particle orientation and that sensitivity decreases when increasing the concentration of magnetite grains. In addition, the rectangular-shaped sample imposed by the rheological device does not affect measures of AMS significantly. The results give reasons to consider that the measure of AMS is an accurate and sensitive method to access the mean local rheological behavior in filled or non-filled systems containing anisotropic tracers.

Keywords Filled polymer · Flow induced orientation · Shear flow

Introduction

Orientation in soft materials remains of great importance as many of the final properties depend on it. For polymer melts, macromolecule orientation leads to higher stiffness and modulus in the flow direction. For filled polymers and especially fiber-reinforced thermoplastics, the filler orientation controls the anisotropy of mechanical properties. This is even true for any filled polymeric systems containing anisotropic fillers or polymer blends with deformable dispersed phase (Kenig 1986; Vincent and Agassant 1986; Bay RS and Tucker 1992; Gupta and Wang 1993). In industrial polymer processing such as injection molding or extrusion, the control of molecular or fillers' orientation is merely achieved by controlling the kinematics of the flow (Karpov and Kaufman 1965; Kaliske and Seifert 1975). As a consequence, strain history can be inferred from the observation of particle orientation or molecular orientation. This is often used in Earth Sciences for which crystallographic fabric of minerals (shape and lattice preferred orientations) are classically used to provide key information on the flow direction and internal deformation of magmatic rocks (Ventura et al. 1996).

The accurate characterization of the overall direction of the filler in a flow is then fundamental. Generally, image analysis subsequent to TEM or MEB observations is used to describe qualitatively or even quantitatively the filler orientation. This method has been extensively used for instance in the case of polymer/glass fiber systems after injection molding (Giroud 2001; Megally 2005). Quantification of the distribution of fiber orientation in the thickness of

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the samples was also performed by surface analysis complemented by three-dimensional measurements by X-ray microtomography. The observation and quantification of orientation made it possible to detect the presence of a core-skin structure, similar to that observed with composites loaded short fibers. Generally speaking, these methods are clearly tiresome to perform, and it remains hard to reach full 3D pattern of the fillers' spatial distribution and orientation.

In order to characterize more precisely the 3D orientation of the filler for a polymer/filler mixture, it would be interesting to use physical properties of fillers such as, for example, magnetic properties. This approach is largely used in Earth Sciences through the anisotropy of magnetic susceptibility (AMS) technique that consists in measuring the AMS of rock specimens, centimetric in size, in order to determine the crystallographic fabric related to the solid-state deformation and sedimentary, magmatic or volcanic flow (e.g., Tarling and Hrouda 1993). The AMS technique overcomes the difficulty and time-consuming nature of crystallographic fabric characterization that are obstacles to systematic studies. AMS is a non-destructive and relatively fast technique that allows to reveal the magnetic fabric of rocks. It is widely accepted that the AMS fabric usually correlates well with the crystallographic fabric developed during magmatic or solid state (e.g., Rochette et al. 1992; Bouchez 1997). For fabric generated by flows, such as basaltic lava flow or dikes, relationships between magnetic and crystallographic fabric are less straightforward because of the low magnetic anisotropy values usually measured. However, recent multi-scale methodological studies (from crystal to lava flow of decametric thickness) on natural lava flows and dikes indicate that AMS technique is also perfectly appropriate to obtain information on flow directions (Bascou et al. 2005; Hastie et al. 2011). In addition, analogical and numerical flow models have also been developed (Fernandez et al. 1983; Dragoni et al. 1997; Arbaret et al. 2000; Cañón-Tapia and Chávez-Álvarez 2004) to better constrain relationship between AMS and crystallographic fabric.

More recently, Kratinová et al. (2006) studied the modeling of flows in structures called “diapir” resulting from the rise of lighter rocks through denser rock. This analog modeling of lava domes uses the AMS signature to map the flow-induced fabric. Results provide interesting insights and help unravel the internal strain pattern during ascent of non-linear viscous materials. Nevertheless, some aspects of relationships between AMS and flow remain to be clarified, and further experiments in which rheological parameters can be rigorously quantified are required.

In this work, we will check the ability and sensitivity of AMS to measure the orientation of magnetic particles during a controlled flow in viscoelastic medium. A set of

experiments using sheared polymer with admixed magnetite grains, micrometric in size, and having a slightly anisometric shape, which served as a tracer of the flow-induced magnetic anisotropy was performed. Experiments were conducted by varying various parameters such as the viscosity of the matrix, the shear, and the concentration of magnetic particles. Moreover, we checked the effect of local flow changes on the magnetic tracer orientation by adding large non-deformable and spherical particles. Two main questions will then be addressed. How much is the sensitivity of AMS versus changes in rheological conditions? Is it possible to use these measurements to get information about orientation in more complex systems?

Experimental section

Materials

The polystyrene used as received is the Lacqrene 1810 that is amorphous polystyrene, in granular form, from the supplier Atofina. Viscosity measurements were performed at 180 and 195 °C. Figure 1 shows the variations of the complex shear viscosity obtained from measurements made in a controlled-stress rheometer from Rheometrics Company (SR5000). Values of the zero shear viscosity are gathered in the Table 1.

Magnetite is from the supplier Prolabo and was used as received. Characterization of the magnetite particles' composition was carried out from the Curie temperature determination using the susceptibilimeter Kappabridge AGICO (KLY-3S and MFK1-FA) coupled with the ovens CS-4 and CS-L which allows measurements of magnetic susceptibility in function of temperature from −192 to 700 °C, under a weak magnetic field (200 A/m) and inert atmosphere (argon gas). Figure 2 shows typical thermomagnetic curves with a Curie temperature of 580 °C. Moreover, at low temperatures, plotting the susceptibility variation with

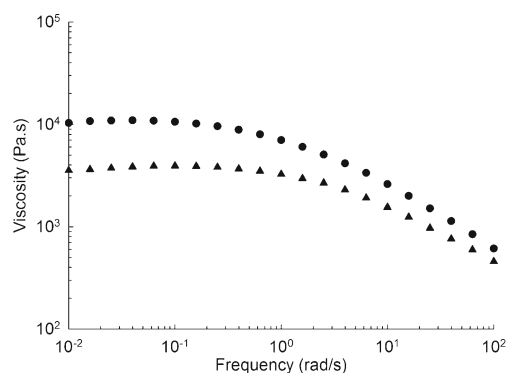


Fig. 1 Complex shear viscosity of polystyrene matrix, as a function of frequency at 180 °C (filled triangles) and 195 °C (filled circles)

Table 1 Polystyrene viscosity at 180 and 195 °C

Polymer	Temperature (°C)	Zero shear viscosity (Pa.s)
Polystyrene	180	10,000
L1810	195	3,500

temperature allowed us to determine the Verwey transition around -163 °C (Fig. 2). Curie temperature and Verwey transition are intrinsic properties dependent only on chemical composition (Titanium content for example), and the observed values confirm that the used magnetic particle is close to pure magnetite (Fe_3O_4).

The magnetite particles have been observed using scanning electron microscopy on dispersed magnetite grains in polystyrene matrix. In order to quantify shape parameters of particles, image analysis has been carried out from the SEM micrographs. As the obtained images were not contrasted enough, some “masks” of SEM micrographs were built (Fig. 3). The distribution of particles in a viscous matrix at low content (5 and 10 wt%, see next paragraph for the mixing conditions) leads to clear pictures where the particles are well separated and dispersed. In addition, it appears qualitatively that the shape of particles is not drastically affected by the mixing. Image analysis gives the average radii weighted by the number of particles, the circularity (C) and the shape ratio (SR). Table 2 and Fig. 4 summarize the results for image analysis and characterization of particle size distribution. Radii are ranged between 0.05 and 10 μm , with a majority of particles having a radius of 0.6 μm and could therefore consist largely of pseudo single domain (PSD) grains because the size of PSD grains of magnetite is commonly considered ranging between larger grains (>20 μm) of multidomain structure and smaller grains (<0.1 μm) of single-domain structure

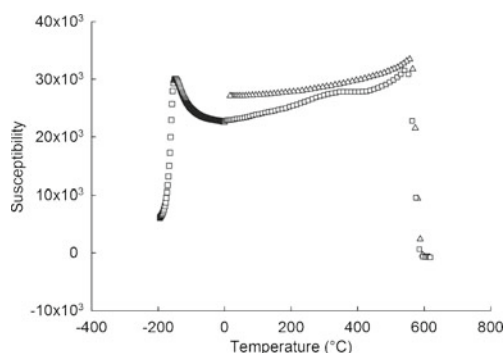


Fig. 2 Variation of the susceptibility of magnetite powder with the temperature (°C), during heating (unfilled squares) and cooling (unfilled triangles)

(Dunlop 2002). Moreover, the use of form factors like the circularity (C) and the shape ratio (SR) shows the shape anisotropy of magnetite particles with a length approximately 1.6 times greater compared to the width. The circularity is defined as follows: $c = 4\pi \times \text{area}/\text{perimeter}^2$. The shape ratio corresponds to the ratio between the long and short axes of the equivalent ellipsoid. The variations in measured shape ratio and circularity show the limits of the image analysis.

Sample preparation

The mixture of polystyrene granules with magnetite particles was performed using a mixer from Thermo Scientific, which is composed of a base device (Polylab) and a measuring cell (Rheomix). Polystyrene in granular form and magnetite in powder form are weighed depending on the desired composition and stirred manually for 1 min. They are then introduced into the preheated mixer. The torque and the temperature variations are monitored and recorded continuously. The mixing speed used was 20 rpm. The mixing time was set to 10 min from the maximum of the torque variation, and the mixing temperature was set to 160 °C.

The flow was generated using a capillary rheometer with a rectangular die having the following dimensions: length $L = 30$ mm, width $W = 10$ mm and thickness $H = 1$ mm. Shear rate in the direction of the thickness was obtained using the following equation:

$$\dot{\gamma} = \frac{12Q}{We^2} \quad (1)$$

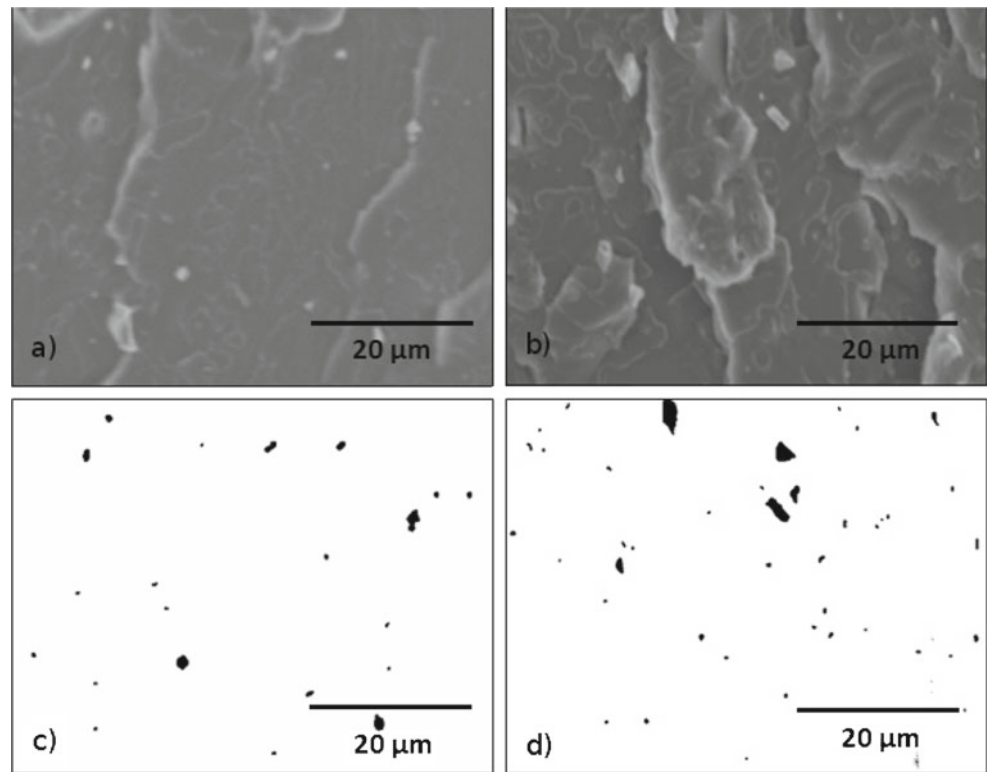
with W and e , respectively, being the width and the thickness and Q being the volumetric flow rate. The non-Newtonian nature of the suspending fluid was taken into account applying the Rabinovitch procedure. Considering the geometry of the die, the shear rate along the width was neglected.

AMS principles and measurements

Magnetic susceptibility K is defined by $M = K \cdot H$, where M is the induced magnetization of the specimen and H is the applied magnetic field. M and H are expressed in amperes per meter and K is dimensionless classically measured at room temperature and low field strength ($<10^3$ A/m).

For a non-isotropic material, the magnetic anisotropy is expressed by a symmetrical second rank tensor. The geometrical representation of this tensor is an ellipsoid defined by eigenvectors and eigenvalues where K_1 , K_2 , and K_3 are

Fig. 3 **a, b** SEM images obtained for the polystyrene/magnetite mixtures (PS/M) at 5 wt% (*left*) and PS/M at 10 wt% (*right*). **c, d** Particles are highlighted after threshold and image analysis



the maximum, intermediate, and minimum principal axes, respectively (Fig. 5).

High intrinsic-susceptibility minerals like magnetite have magnetic anisotropy that is controlled dominantly by particle shape. Thus, the magnetic susceptibility ellipsoid displays similar orientation and aspect ratio as the grain itself. For a sample containing a population of very high intrinsic susceptibility grains with dominant shape anisotropy (Fig. 5), the resulting AMS represents a composite of grain shape and preferred orientations (e.g., Grégoire et al. 1998). The mean bulk susceptibility, eccentricity, and shape of the AMS ellipsoid can be characterized by various parameters (Jelinek 1981). The most common are defined in the Table 3 below.

AMS measurements were performed using the susceptibilimeter Kappabridge (KLY-3S and MFK1-FA; AGICO company) with an applied field of 200 A/m and frequency

Table 2 Number of particles (N), average radii (R_n) weighted by the number of particles, circularity (C), and shape ratio (SR) for the polymer/magnetite blends at 5 and 10 wt %

	N	R_n (μm)	C	SR
PS/M-5 wt%	29	0.60	0.89	1.58
PS/M-10 wt%	37	0.60	0.84	1.73

of 976 Hz. In the used spinning method, the specimen rotates with a low speed of 0.4 rps inside the coil, around the three axes consecutively. The specimen susceptibility is measured in 15 different orientations, and from these values, six independent components of the susceptibility tensor and statistical errors of its determination are calculated. This tensor carries information only on anisotropic components of the specimens. So as to obtain a complete susceptibility tensor, one additional measurement of bulk susceptibility must be done.

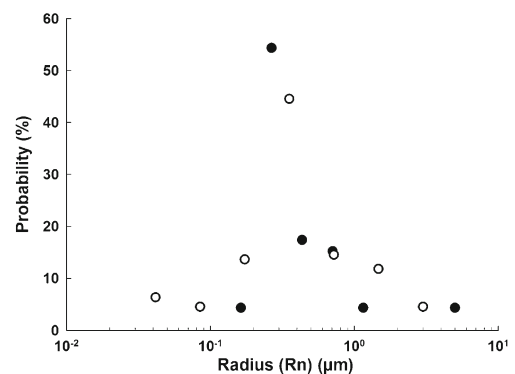


Fig. 4 Distribution of particles' size of magnetite powder dispersed in polystyrene obtained for the polystyrene/magnetite mixtures at 5 wt% (*filled circles*) and PS/M at 10 wt% (*unfilled circles*)

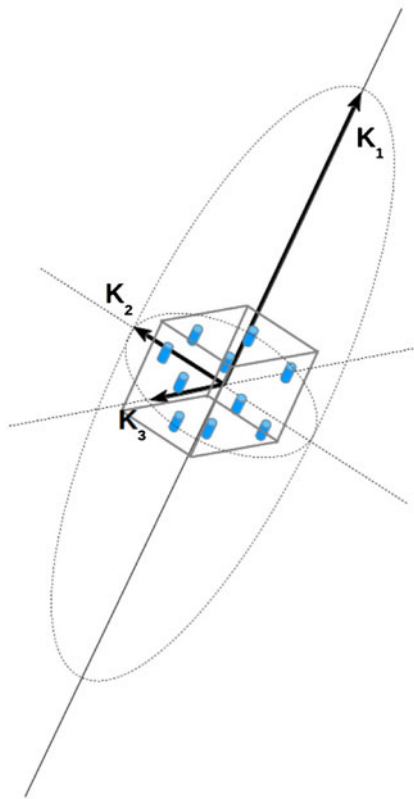


Fig. 5 Ellipsoid of magnetic susceptibility (dot line) around the cubic sample containing anisotropic and a population of oriented particles

The holder of this equipment is commonly sized for cylinders with diameter: 25.4 mm and length: 22 mm or cubes ($20 \times 20 \times 20$ mm). We therefore used a sample holder made in non-magnetic material (high-density polyethylene) in order to perform AMS measurements on very small samples obtained from flow in a capillary rheometer (rectangular samples $20 \times 10 \times 2$ mm). It is worth noting that due to die swelling, the thickness of the final sample is larger than the dimension of the flow apparatus.

Table 3 AMS parameters with $K_1 \geq K_2 \geq K_3$. The shape parameter T that characterizes the shape of the AMS ellipsoid and hence the type of magnetic fabric varies from -1 (prolate shape) to 1 (oblate shape), from Jelinek (1981)

Parameters	Formulas
Degree of anisotropy	$P = K_1/K_3$
Shape parameter	$T = (2\ln K_2 - \ln K_1 - \ln K_3) / (\ln K_1 - \ln K_3)$
Magnetic lineation	$L = K_1/K_2$
Magnetic foliation	$F = K_2/K_3$
Mean susceptibility	$K_m = (K_1 + K_2 + K_3) / 3$

Results and discussion

Effect of the sample shape on AMS measurements

As already mentioned, AMS measurements are commonly performed on cubic or cylindrical specimens with relatively low shape ratio. This prevents possible errors generated by the shape of specimens. Samples obtained from the flow of polystyrene/magnetite (PS/M) suspensions through the capillary rheometer do not follow these conditions. An important dimensional anisometry of the samples is observed. Therefore, before evaluating the effect of the flow and the matrix viscosity on the variations of the magnetic susceptibility tensor induced by particles orientation, we will first check the impact of the sample shape on the AMS measurements.

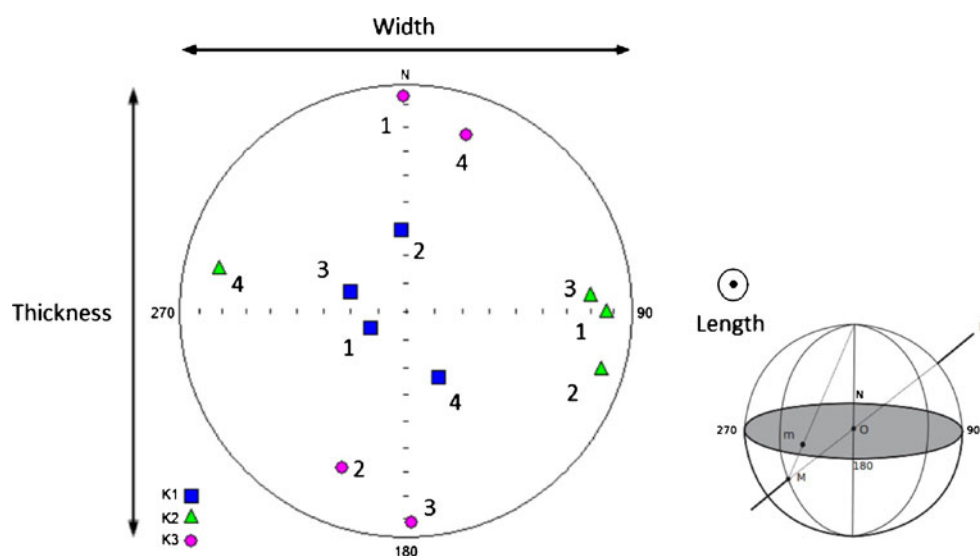
Firstly, the orientation state of magnetite particles after mixing has been investigated. Because of the high distributive nature and complexity of the flow in the internal mixer, random orientation of the particles is expected. This has been checked using cubic samples of about 7-mm sides directly carved in the bulk. The results show that no specific orientation is observed. Therefore, before the flow across the rectangular die, the anisotropic particles are randomly oriented in the polystyrene matrix.

In a second step, AMS measurements have been applied on rectangular samples (length = 2 cm, width = 1 cm, thickness = 1 mm) directly carved in the bulk of the mixture obtained at the output of the mixer. In addition, rectangular samples were cut in half lengthwise, to amplify the shape ratio in the direction of the length.

Figure 6 shows clearly that the axis of maximum susceptibility (K_1) tends to be located in the direction of the length of the sample. This is observed whatever the amount of magnetite in the sample. Then, an effect of the sample shape on the orientation of the ellipsoid can be suspected. Moreover, Table 4 shows that, for the samples cut in half (samples 3 and 4), K_3 does not vary, K_1 increases, and K_2 decreases.

However, neither K_2 nor the degree of anisotropy P are proportional to the sample shape ratio. Indeed, in Table 4, it appears that a decrease of about 50 % of the width of a rectangular sample implies only a decrease of 15.15 % of the value of K_2 for the mixture at 5 wt% and a decrease of 5.5 % of the value of K_2 for the mixture at 10 wt%. The effect of the sample shape exists but it is weak as indicated by the very close values of the anisotropy parameter (P) before and after cutting (P values variation lower than 10 %). Actually, small variations around an average sample size will not affect dramatically the values of the magnetic susceptibility anisotropy. We then suggest that in order to be quantitative, AMS measurements have to be performed and compared with samples with similar dimensions.

Fig. 6 Projection in the lower hemisphere of K1, K2, and K3 principal magnetic susceptibility axes measured on rectangular samples obtained at the output of the polystyrene/magnetite mixer: 1 = PS/M–5 wt%, 2 = PS/M–10 wt% and rectangular samples cut in half: 3 = PS/M–5 wt%, 4 = PS/M–10 wt%. On the right is the illustrated principle of projection in the lower hemisphere. The *D* axis (projected axis) passing through *O* intersects the lower hemisphere in *M*. *m* is the projection of *M* in the equatorial plane (gray area)



The relative weak effect of the sample shape is confirmed by the similar orientations of K1, K2, and K3 axes on both cubic and rectangular samples obtained after flow across the rectangular die (Fig. 7). The cubic sample of about 1-mm sides was prepared from the previous rectangular sample (5 wt%, 180 °C and 10,000 Pa.s). Projections of principal susceptibility axes show that the K1 axis orientation is parallel to the flow direction while the K3 axis is perpendicular to the flow direction (along the thickness of the sample). The orientation of the principal susceptibility axes is therefore consistent with the flow direction. Results are very similar to those obtained for the rectangular sample. The conclusions are twofold. First, we confirm the weak influence of the sample shape compared to the effect of developed preferred orientation of particles. Secondly, these results show the sensitivity of AMS even for very low sampled volumes.

Effect of magnetite concentration, matrix viscosity, and shear

This section is devoted to the ability of AMS to reveal particle orientation for various rheological conditions of flow.

Table 4 Values of K1, K2, K3, and *P* measured for rectangular samples

	K1	K2	K3	<i>P</i>
PS/M-5 wt%	1.19	1.13	0.67	1.78
PS/M-5 wt% (cut in half)	1.25	0.96	0.67	1.86
PS/M-10 wt%	1.18	1.08	0.85	1.39
PS/M-10 wt% (cut in half)	1.20	1.02	0.84	1.43

As explained in the previous section, two concentrations of magnetite (5 and 10 wt%), two viscosities of the PS matrix (10,000 and 3,500 Pa.s), and two shear strains (5.5 and 0.55 s⁻¹) have been used for this study.

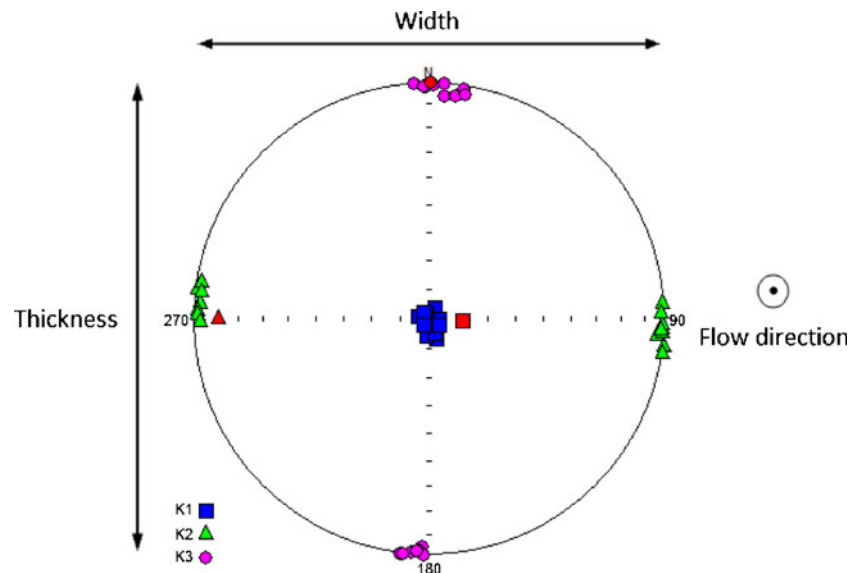
Figure 8 gathers the results for all experiments and represents the degree of anisotropy (*P*) as a function of the average susceptibility (*K_m*) normalized by the volume of the sample which captures the impact of the fraction of magnetic particles.

Firstly, the AMS measurement seems to be more sensitive to variation of the rheological conditions for low concentrations (5 wt%). AMS is able to discriminate significantly between the experiments under different viscosities and shear rates. For a given concentration and the same shear, the degree of anisotropy *P* increases as the viscosity increases.

Moreover, at the same viscosity and for low concentrations (5 wt%), the degree of anisotropy increases as the shear rate increases. In the same way, as the shear rate increases, we observe the same variation between the values of *P*, whatever the viscosity value. Qualitatively, these variations can be explained considering the history of stress and strain during the flow. For instance, at the entrance of the die, the intensity of the elongational flow depends on the viscosity of the fluid. Moreover, at the output of the die, the swelling observed induces relaxation of the particles' orientation driven by the relaxation of the viscoelastic suspending fluid and consequently its viscosity. Particles' orientation state is not governed by the rotational Peclet number (particles are not Brownian objects). This phenomenon illustrates why from AMS measurements, it appears that orientation is greater for the highest viscosity whatever the shear stress.

Finally, as expected, it appears clearly that the average susceptibility *K_m* increases proportionally with the

Fig. 7 Projection in the lower hemisphere of K1, K2, and K3 axes, with the right (imposed) flow direction of rectangular samples (for all magnetite amount and flow conditions) prepared after the flow across a rectangular die and the cubic sample PS/M at 5 wt%, 10,000 Pa.s and 5.5 s⁻¹ (red)



magnetite concentration. To summarize, for low magnetite concentration (5 wt%), AMS is sensitive enough to reflect changes in viscosity and shear rate. The sensitivity of AMS decreases when increasing the magnetite concentration. It could be due to the effect of magnetic interactions between the particles for the relative high magnetite concentration. When an external magnetic field is applied on the sample (in the solid state), magnetostatic interactions between the particles could arise out of the nearness of the dipoles. These interactions could change the response of each par-

ticle and so the global response of the sample. This is in agreements with recent results of the models of Fanjat’s et al. (2012a, b) showing that when magnetic interactions increase, the anisotropy ellipsoid tends towards a sphere introducing noise in the magnetic signature with a consequent decrease in the degree of anisotropy and development of abnormal magnetic fabrics.

Implication in the understanding of AMS signature for more complex systems

In order to be closer to the behavior and composition of natural systems (such as lava or dykes) that represent relatively complex medium with various minerals, the effect of adding a filler on the orientation of magnetite particles was studied. The selected filler is the silica from the supplier Potters–Ballotini. It was used in powder form and particles have an average size of 30–50 μm. For this, AMS measurements were made on samples obtained by the flow of the mixture PS/M2-5 %/silica-20 % through the capillary rheometer at two different viscosities (10,000 and 3,500 Pa.s) and at 5.5 s⁻¹. The concentration of silica is given by mass. Table 5 shows that, at the measurement time, the orientation state in the presence of silica is lower. Indeed, a decrease in the

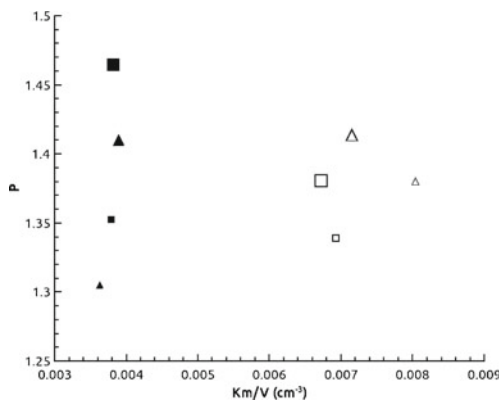


Fig. 8 Curve representing the degree of anisotropy depending on the average susceptibility normalized by the sample volume, for samples obtained at different conditions: 5 wt%, 10,000 Pa.s, 5.5 s⁻¹ (big filled square), 5 wt%, 10,000 Pa.s, 0.55 s⁻¹ (big filled triangle), 5 wt%, 3,500 Pa.s, 5.5 s⁻¹ (small filled square), 5 wt%, 3,500 Pa.s, 0.55 s⁻¹ (small filled triangle), 10 wt%, 10,000 Pa.s, 5.5 s⁻¹ (big unfilled square), 10 wt%, 10,000 Pa.s, 0.55 s⁻¹ (big unfilled triangle), 10 wt%, 3,500 Pa.s, 5.5 s⁻¹ (small unfilled square), 10 wt%, 3,500 Pa.s, 0.55 s⁻¹ (small unfilled triangle)

Table 5 Degree of anisotropy P obtained for mixtures PS/M_5 wt% in each case

	P without silica	P with silica
10,000 Pa.s, 5.5 s ⁻¹	1.53	1.12
3,500 Pa.s, 5.5 s ⁻¹	1.38	1.25

degree of anisotropy P is observed with the addition of silica.

This result can lead to rapid understanding of AMS signature such as those encountered in lava flows. The polymineralogic content of natural basaltic flows could then favor low magnetic anisotropy despite high shear rates and viscosity. This could also explain low anisotropy degree generally measured in lava flow (<10 %, Hrouda 1982) despite clear evidence of deformation structures such as crystallographic preferred orientations or bubble alignments. The presence of micrometric fillers strongly disturbs the local flow in the suspending fluid as already showed by Le Meins et al. (2003).

Conclusion

AMS measurements performed on samples obtained from the flow of PS/M mixtures, through the capillary rheometer and under different conditions of shear rate and viscosity, showed that for all samples, the orientation of the magnetic susceptibility K1 axis is parallel to the flow direction and also along the length of the sample, and the K3 axis is oriented along the thickness. This orientation of principal susceptibility axes is consistent with the flow direction and particle orientation. The impact of sample shape on the susceptibility tensor has been checked, and it was shown that the flow-related preferred orientation of particles overcomes this effect. Then results show that the AMS is an accurate and sensitive method to inform on particle orientation and distribution. Very few quantities of anisotropic magnetic particles are sufficient to reveal the magnetic fabrics. Such particles can be used as tracer of the local rheological conditions. The effect of adding a filler (silica) produces a decrease of anisotropy degree. However, the magnetic anisotropy remains higher than natural values. Further studies that incorporate magnetite grains with various shape factor and silicate particles with different geometries and densities should allow better modeling of the AMS in natural flows. Admixture of magnetic tracers in polymer blends (either in the matrix or the dispersed phase) will be an opportunity to access local rheological behavior of these systems.

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