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A SIMPLE SAMPLE-MOUNTING METHOD FOR RANDOM POWDER X-RAY DIFFRACTION

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Abstract—This paper describes an improved simple, sample-mounting method for random powder X-ray diffraction (XRD), namely the razor tamped surface (RTS) method, which prepares a powder mount by tamping the loose powder with the sharp edge of a razor blade. Four kaolinites and a quartz powder were used to evaluate the RTS method by quantifying the degree of orientation in the sample mounts using orientation indices. Comparisons between the RTS and other published simple methods, consisting of front loading, back loading and side loading, indicate that the RTS method produces minimum packing density and minimum preferred orientation in the powder mounts of all five samples. The quartz powder used in this study does exhibit a tendency to preferred orientation. The mechanism by which the RTS method reduces preferred orientation is examined by comparing the width of the sharp blade edge with the size of clay particles. The advantages and disadvantages of the RTS method are also discussed.

Key Words—Crystallinity, Kaolinite, Preferred Orientation, Quartz, Random Powder, Sample Preparation, X-ray Diffraction.

INTRODUCTION

X-ray diffraction (XRD) is a very powerful technique for both qualitative and quantitative mineralogical analysis. In many cases, a random powder mount is necessary for the characterization of detailed mineralogy. For example, random powder must be used to differentiate completely dioctahedral from trioctahedral subgroups of clay minerals by 060 reflections (Brown and Brindley, 1980; Moore and Reynolds, 1997). In fact, achieving truly random orientation has been a long-term goal in quantitative XRD analysis and "one of the long-standing problems" (Bish and Reynolds, 1989). However, just as perfect orientation can never be obtained, complete randomness in a powder mount is equally elusive, especially for platy clay particles prone to preferential orientation.

Significant effort has been made to develop robust and simple techniques to achieve random orientation in a sample mount. All kinds of methods fall into two broad groups: (1) techniques without pre-processing the dry sample powder; and (2) techniques which require pre-treatment of the powder, adding filling materials to the powder, or adding adhesives to the sample holder. The former consists of several simple methods, including front loading, back loading and side loading, in which the powder is pressed or tamped into a cavity on the sample holder from various directions referenced to the examined sample surface. The latter includes: (a) pre-treating the sample powder to form small agglomerates containing randomly oriented particles, such as freeze drying (Moore and Reynolds, 1997) and spray drying (Jonas and Kuykendall, 1966; Hughes and Bohor, 1970; Calvert *et al.*, 1982; Hillier, 1999); (b) adding adhesives to the sample holder so that particles stick to the surface with random orientation, such as adding gelatin to the sample holder (H.L. Barwood, pers. comm.) or smearing grease on the slide (Brown and Brindley, 1980); (c) adding foreign filling materials to the sample powder to reduce preferred orientation, *e.g.* embedding the sample in a thermoplastic organic cement (Brindley and Kurtossy, 1961; Hinckley, 1963) or mixing with powdered cork (Wilson, 1987) or glass beads (Jenkins *et al.*, 1986); and (d) making a clay suspension in acetone instead of water to reduce preferred orientation by decreasing the surface tension of the suspension fluid (Paterson *et al.*, 1986). As pointed out by Brown and Brindley (1980), these more elaborate methods are tiresome for routine work. Certain methods require special equipment which may not be available commercially. In addition, as one problem is solved, another problem arises. For example, embedding clay in an organic cement contaminates the sample and reduces the mass absorption coefficient of the mixture, resulting in significant shifts of peak position in a thick sample, known as "transparency effects" (Klug and Alexander, 1974; Bish and Reynolds, 1989). After reviewing a series of techniques for preparing a random powder mount, Brindley (1980) concluded that side packing was probably the best simple technique available.

The kaolinites are typically 0.1 to ~4 μm wide platy particles (Mitchell, 1993) and generally tend to orientate preferentially during sample packing. Therefore, they are often used to evaluate sample-mounting methods. In fact, Brindley and Kurtossy (1961) found in a

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quantitative analysis that the degree of orientation was high for well crystallized kaolinites and diminished for those which were poorly crystallized. To quantify the degree of preferred orientation, they proposed an orientation index (OI) for clay minerals as

$$\mathrm{OI} = \frac{\text{Intensity of a suitable basal reflection, } I_{00l}}{\text{Intensity of a non-basal, prism reflection, } I_{060}}$$

in which l = 1 or 2 and I is the intensity of a reflection. Orientation increases I_{001} and I_{002}, but reduces I_{060}. As basal plane orientation increases, OI increases rapidly. Therefore, the ratios of I_{001}/I_{060} and I_{002}/I_{060} give a quantitative measurement of orientation. Though another prism reflection 020 gives diffuse scattering for kaolinites with layer stacking disorder (Brindley and Kurtossy, 1961), it occurs in the same angular range as the 001 and 002 reflections. Therefore the ratio of I_{002}/I_{020} can still be used as an orientation indicator for less disordered kaolinites.

Quartz grains generally exhibit conchoidal fracture and occur as anhedral particles. It is commonly believed that quartz shows little or no tendency to preferred orientation. Therefore, quartz is seldom used as a material to assess sample mounting methods. However, Eslinger *et al.* (1973) found that the ratio of the intensities of the two strongest peaks 100 and 101, *i.e.* I_{100}/I_{101}, obtained from thin suspensions sedimented onto glass slides varied from 0.12 to 28.5. They attributed the variation to preferred orientation of authigenic quartz, which tends to form well developed (100) prism faces. Furthermore, Krinsley and Smalley (1973) showed that the relative magnitude of fracture and cleavage in quartz is a function of grain size. For grains >500 μm, fracture predominates over cleavage, while cleavage is dominant in those <50 μm in diameter. Therefore, some quartz powders of a certain grain size and origin may tend to a preferred orientation, though it is much less prone to have preferred orientation than kaolinite.

This paper presents a mechanical technique for preparing random powder mounts, which does not require any pre-processing of the dry sample powder. It aims to provide a simple method readily available to any laboratory. Since a razor blade is used to tamp the loose powder to form a special packing surface, it is named the razor tamped surface (RTS) method.

EXPERIMENTAL

Materials

To evaluate the RTS method, both kaolinites and a quartz powder were used to prepare powder mounts. Four essentially pure kaolinites varying in crystallinity, including two from The Clay Minerals Society Source Clay Repository, namely KGa-1b (Washington, Georgia) and KGa-2 (Warren, Georgia), and two of unknown origin from the MIT Geotechnical Laboratory which are labeled as Peerless #2 and Grim Calibration (K-GC), respectively, were used. Since KGa-1b and KGa-2 are widely used, results from this research can be compared with others in the literature. Due to its low level of crystal defects, KGa-1b was chosen to measure the reproducibility of the RTS method. Another sample material used was a pure quartz powder with unknown origin, which was wet ground to <44 μm before analysis.

Sample mounting methods

Random powder mounts were prepared for XRD using the three widely used sample mounting methods (*i.e.* front loading, back loading and side loading) and the RTS method. Here front loading means simply pressing the powder into the cavity with a frosted glass slide by applying a normal force and shear motion to make the surface of the powder mount smooth and flat. The back-loading method described in Klug and Alexander (1974) was adopted here. For side loading, one side of the window in the holder was removed so that powder could be loaded from that side. The procedures of McMurdie *et al.* (1986) and Moore and Reynolds (1997) were followed to load the powder from the side. During this investigation, it was found that side-packing the powder with 3 to 4 layers into the holder helped to make a more homogeneous mount. Therefore, during side loading, the sample was packed with several layers. It is worth pointing out that, during this investigation, a frosted glass slide was placed facing the powder mount for both side loading and back loading.

The 'standard' sample holder provided by the manufacturer was used in the RTS method. It is a rectangular Al plate with dimensions $50.2 \times 32.2 \times 1.6$ mm containing a rectangular window of $20.0 \times 18.0 \times 1.6$ mm. Preliminary examination found that the powder mount was relatively loose and tended to fall out of the cavity in a vertical-axis diffractometer if a clean glass slide was used on the back of the window to support the powder. To give the powder more adhesion to the holder, a piece of double-sided sticky tape was used to hold the glass slide to the sample holder. Furthermore, it has been found that the powder mount gained more stability if the four sides of the cavity were tapered toward the back surface (Figure 1).

The procedures of mounting the powder by the RTS method are as follows (Figure 1):

(1) Cut from a microscope slide a piece of glass slightly larger than the empty window in the Al holder. Cover it with a piece of double-sided sticky tape, and then stick it to the back of the window to form a cavity with front surface open.

(2) After mixing on a clean glass plate with a razor blade to remove any pre-existing orientation, the powder is transferred without any free falling height into the cavity. Add more powder until it is 3–5 mm deep on the holder surface.

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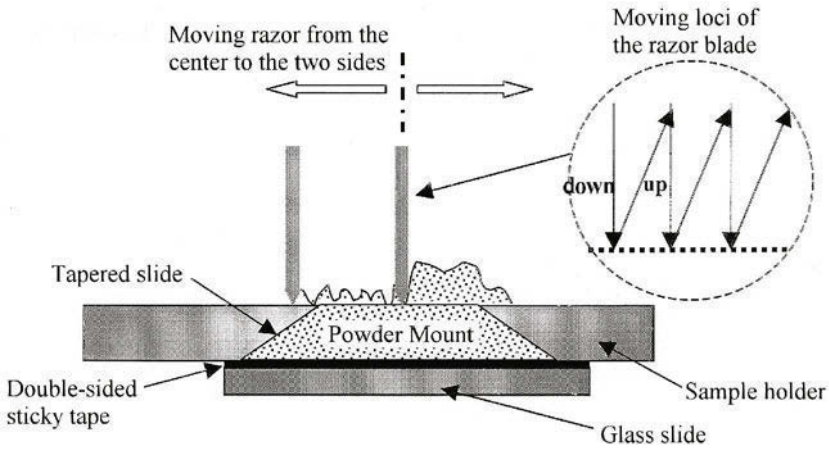

Figure 1. Illustration of the sample holder and the RTS method (not to scale).

(3) Chop the powder gently (40–50 times) in random directions using the sharp edge of a razor blade to cause initial compaction and more uniform packing in the cavity. The blade must be held vertical (in both up and down motion) to eliminate any shearing motion on the holder surface.

(4) Tamp the powder again from the center to the two sides, following a different motion path, in which the blade is pushed downward straight and lifted upward at an angle of 30–45° to the vertical. Some excess powder can be moved away from the cavity during this step.

(5) Blow across the surface of the holder to remove the loose surplus by keeping the holder horizontal at a distance of ~0.5 m. Do not try to remove all loose surplus at once. Never slice off the surplus powder with a razor blade.

(6) Repeat steps 4 and 5 in alternate directions to remove all loose surplus until a flat and relatively smooth surface appears on the powder mount.

X-ray powder diffraction

The XRD patterns were obtained in a Rigaku Rotaflex 180 mm diffractometer with a graphite-diffracted beam monochromator, using CuKα radiation generated at 18 kW (60 kV, 300 mA), a continuous scan range of 2–64°2θ, a scan speed of 1°2θ/min, and a step size of 0.02°2θ. All peak positions of kaolinites and quartz were identified with the help of the commercial software, JADE. All peak intensities were determined with the help of JADE, based on the net peak height (*i.e.* excluding background).

Environmental scanning electron microscope

To investigate the mechanism by which the RTS method reduces preferred orientation, the sharp edge of a razor blade was examined microscopically using a FEI/Philips XL30 FEG environmental scanning electron microscope (ESEM), under which the dimensions of the sharp blade edge were measured accurately.

RESULTS AND DISCUSSION

Degree of orientation

The diffraction patterns of Peerless #2 are shown in Figure 2 as an illustrative example for the four kaolinites, while Figure 3 shows the diffraction patterns of the quartz. It is worth pointing out that the 020 peak height of kaolinites is measured based on the general background, while the background for 002 is the diffuse scattering line of 020 (Figure 2). Table 1 summarizes all the orientation indices for the four kaolinites together with those estimated from publications in the literature, while Table 2 lists the results for the quartz powder.

For the four kaolinites, it is first noticed that RTS method gives the smallest OIs, I_{002}/I_{020}, I_{002}/I_{060} and I_{001}/I_{060}. The only exception is that KGa-2 has nearly the same I_{002}/I_{060} for the RTS method (3.67) as the side-loading method (3.66). This may be attributed to the high level of defects in KGa-2. Table 1 also gives the measured kaolinite Hinckley Index (HI) proposed by Hinckley (1963). It appears that the HI of each kaolinite is relatively independent of sample-mounting techniques. However, as the HI decreases from K-GC (HI = 1.22) to KGa-2 (HI = 0.16), OI generally decreases. This is consistent with the results obtained by Brindley and Kurtossy (1961). Peerless #2 (HI = 0.77) is the exception, and this may be attributed to the effect of particle size. Moreover, for each kaolinite, the difference in the OI values between the RTS method and others (side loading and back loading) increases with increasing HI. This may be attributed to the fact that well crystallized kaolinites show a greater tendency to preferred orientation. It also indicates that back loading and side loading do not effectively reduce the preferred orientation for well crystallized kaolinites. As an example to illustrate the preferred orientation caused by front loading, the OI value, I_{002}/I_{020}, of Peerless #2 obtained by front loading is also included in Table 1,

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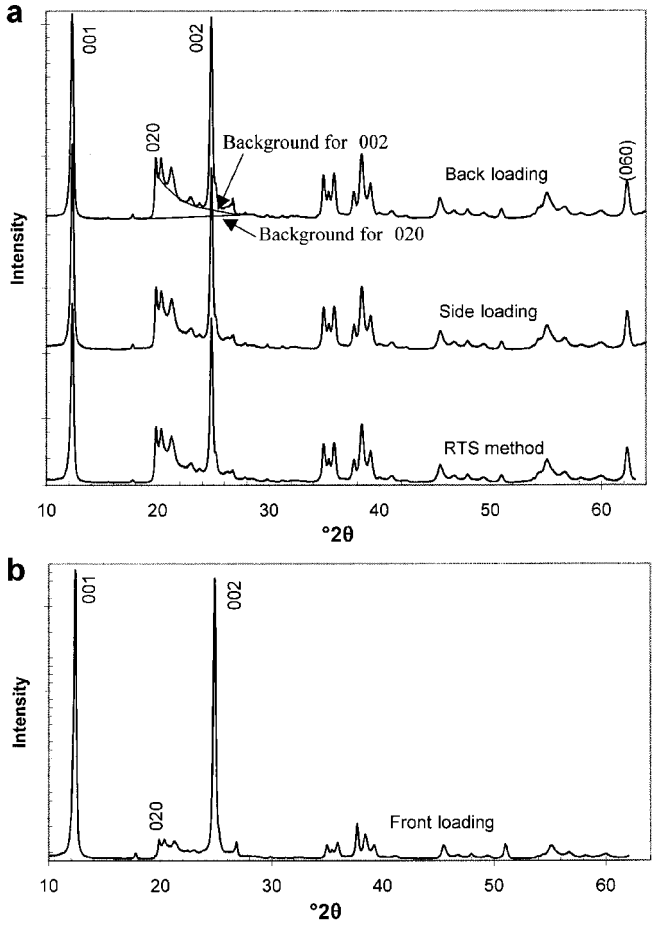

Figure 2. Diffraction patterns of Peerless #2 powder mounts packed by different methods.

which is nearly five times greater than that obtained by other loading methods. It is well known that front loading introduces significant preferred orientation for platy particles.

To evaluate the new method in a different way, Table 1 also lists the OI values estimated from the published diffraction patterns in the literature for the two source clays KGa-1b (or KGa-1) and KGa-2. For KGa-1b, the RTS method has a minimum I_{002}/I_{020} = 2.12, compared with OI by side loading (2.67 for particles <44 μm, Pruett and Webb, 1993) and OI by freeze drying (3.73, Wang *et al.*, 1998). For KGa-2, the RTS method gives I_{002}/I_{020} = 1.95, while spray drying (Hillier, 1999) gave 1.86. However, since the latter was obtained from a 1:1 mixture of KGa-2 and corundum which is usually used as a non-orientating filler material to reduce preferred orientation of clay particles, the contribution of corundum to the reduction of orientation is unknown. Nevertheless, the RTS method achieves a small OI, which is much closer to that obtained by spray drying.

Though Brindley and Kurtossy (1961) gave the calculated OIs for the kaolinite as I_{001}/I_{060} = 3.90, which is greater than that of KGa-1b (3.51) and KGa-2 (3.80) obtained by the RTS method, and I_{002}/I_{060} = 2.12, which is smaller than that obtained by RTS method, they also pointed out that "the importance of the calculated ratios must not be over-exaggerated". In fact, the structure of kaolinites is highly complex as a result of a large number of stacking defects introduced during crystal growth or formation. Therefore, a single calculated diffraction pattern based on the ideal structure is not representative of all kaolinites with different structural disorder and particle size. In other words, for any given degree of orientation, different kaolinites have different values of OIs, as shown in Table 1.

For the quartz powder, the OI, I_{100}/I_{101}, decreases monotonically from 0.271 obtained by the RTS method to 0.256 obtained by the front-loading method (Table 2), indicating that the RTS method gives the greatest reduction of orientation. This agrees well with the common understanding that side loading is better than back loading which in turn is better than front loading. The results also prove that the RTS method can reduce preferred orientation in a quartz powder as well as in kaolinites.

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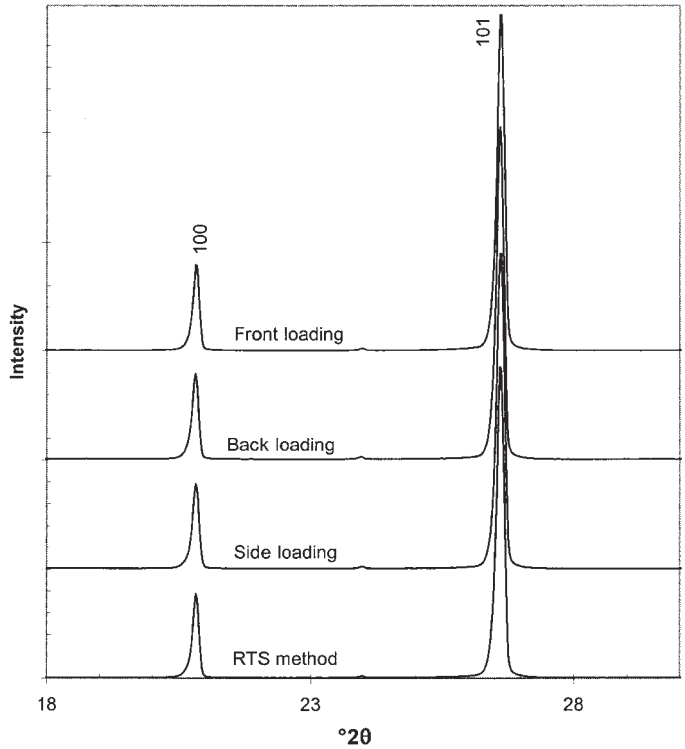

Figure 3. Diffraction patterns of quartz powder mounts prepared by different methods.

Table 1. Comparison of packing density and orientation indices for the four kaolinites.

Kaolinite sample	Mounting method	Packing density (g/cm^3)	Orientation indices I_{002}/I_{020}	I_{002}/I_{060}	I_{001}/I_{060}	Crystallinity index (CI)
K-GC	RTS method	0.19	2.44	5.07	6.32	1.22
	Side loading	0.64	3.29	5.53	6.63	1.30
	Back loading	0.76	3.96	6.55	7.06	1.31
KGa-1b	RTS method	0.45	2.12	3.38	3.51	0.94
	Side loading	0.55	2.15	3.56	3.97	0.94
	Back loading	0.64	2.15	3.65	3.76	0.95
	Side loading [1]		2.67	4.36	4.55	1.09
KGa-1	Side loading [1]		4.48	6.28	8.57	1.03
	Freeze drying [2]		3.73			1.11
Peerless #2	RTS method	0.37	2.79	4.72	5.47	0.77
	Side loading	0.67	2.88	4.77	5.62	0.75
	Back loading	0.67	3.15	5.53	5.81	0.73
	Front loading	0.96	14.16			0.64
KGa-2	RTS method	0.34	1.95	3.67	3.80	0.16
	Side loading	0.41	2.00	3.66	4.00	0.15
	Back loading	0.48	2.08	3.79	3.97	0.18
	Side loading [3]		2.36			
	Side loading [4]		2.36			
	Freeze drying [5]		3.02			
	Spray drying [5]		1.86			

[1] Pruett and Webb (1993), for the <44 μm fraction; [2] Wang *et al*. (1998); [3] Artioli *et al*. (1995); [4] Aparicio and Galán (1999) (may not be KGa-2); [5] Hillier (1999)

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Table 2. Packing density and orientation index of quartz powder mounts.

Mounting method	Packing density (g/cm^3)	Orientation index I_{100}/I_{101}
RTS method	0.73	0.271
Side loading	0.97	0.268
Back loading	1.15	0.257
Front loading (pressed)	1.35	0.256

Sample packing density

As shown in Tables 1 and 2, for the four different kaolinites and a quartz powder, the RTS method always produces the smallest packing density in all powder mounts, while front loading gives the greatest density. For example, the density of the powder mount of Peerless #2 prepared by front loading is 2.5 times greater than that by the RTS method. Apparently, as the powder is compressed from one direction, the platy-shaped clay particles tend to orientate perpendicular to the compression direction. The more the powder is compressed, the higher the degree of orientation the particles have. Therefore, the packing density of the powder mount is an indirect indicator for preferred orientation. Due to the smallest packing density, the RTS method gives minimum preferred orientation in a powder mount.

However, some may question that the packing density is so low that the powder mount may exhibit the so-called 'transparency effects', which can cause potentially significant shifts in peak positions and changes in widths of the observed profiles. Clearly, the absorption characteristics of the sample itself play a more important role than the packing density. Careful examination of the diffraction patterns shown in Figures 2 and 3 found that no measurable shifts of peak positions were detected between different loading methods (*e.g.* RTS, side loading and back loading). Likewise, significant peak broadening was not observed. These observations indicate that the RTS method does not cause significant 'transparency effects' for most silicates, at least for the kaolinites and quartz used in this study. As pointed out by Bish and Reynolds (1989), a thin sample on a non-diffracting substrate is recommended over a cavity mount for accurate measurement of peak positions. The common goal of a cavity mount of random powder is to measure the peak intensity accurately. Therefore, relatively small shifts of peak positions are usually acceptable if accurate measurement of peak intensity is the goal.

Discussion

To assess the reproducibility, Table 3 shows the packing density and OIs of 10 random powder mounts of KGa-1b prepared by the RTS method. Although the density shows a certain variation between different preparations, its values are smaller than those obtained by other methods (side loading and back loading). Likewise, this observation applies to the three OIs. The mean and standard deviations of these parameters are also shown in Table 3.

To investigate the mechanism by which the RTS method minimizes preferred orientation, the sharp edge of a razor blade was examined under ESEM. Although conceptually the thickness of the blade edge is zero, careful measurement under ESEM found that it is ~0.5 μm thick. As pointed out previously, typical kaolinite particles have sizes ranging from 0.1 to 4 μm. This helps to explain why the RTS method is effective in reducing preferred orientation. During packing, when a platy clay particle greater than the blade edge is compressed by the blade edge, the particle will not move vertically with the blade and hence not orientate perpendicular to the compressing direction. In fact, the sharp blade edge is capable of causing rotation of platy particles. For comparison, if a tool having a flat surface and dimension much greater than the size of the particles is used to tamp the powder, then all particles tend to orientate parallel to the surface of the compressing tool. For those particles smaller than the thickness (*i.e.* ~0.5 μm) of the blade edge, it may be argued that the razor blade is not as effective in reducing preferred orientation as for the bigger particles. However, particles <0.5 μm have much less tendency to develop preferred orientation (Bish and Reynolds, 1989).

With respect to the surface of the powder mount and compaction direction caused by razor tamping, the blade edge introduces complex localized two-dimensional particle motion (Figure 4), resulting in various compaction directions around the blade tip and hence significant reworking in the near-surface layer. Since the powder is first packed randomly by tamping along various directions with a blade, a conceptually true random orientation may be achieved in the near surface layer.

In addition to improving random orientation, the RTS method solves several practical problems encountered by side-loading, listed as follows:

Table 3. Orientation indices and packing density of KGa-1b powder mounts prepared by the RTS method.

Test no.	Density (g/cm^3)	Orientation indices I_{002}/I_{020}	I_{002}/I_{060}	I_{001}/I_{060}
1	0.514	2.121	3.384	3.515
2	0.512	2.141	3.435	3.470
3	0.489	2.139	3.470	3.473
4	0.480	2.126	3.393	3.500
5	0.462	2.131	3.568	3.627
6	0.450	2.105	3.362	3.571
7	0.450	2.117	3.523	3.667
8	0.515	2.121	3.399	3.599
9	0.544	2.140	3.454	3.554
10	0.549	2.113	3.341	3.462
Mean	0.496	2.125	3.433	3.544
Standard deviation	0.0361	0.012	0.072	0.072

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(1) The RTS method is simple and requires no special tools or pre-treatment of the sample powder. No special practice is required to be familiar with the technique. In fact, the powder mount prepared by the RTS method has reproducible packing density even for different operators since the powder cannot be compressed further when the blade hits the surface of the Al holder. However, it requires some practice to control the packing effort for side loading, as pointed out by Moore and Reynolds (1997). Different compressing force results in different packing density. Moreover, if the powder is compressed too hard, the surface of the powder may bulge upward after the glass slide is removed, making it impossible to align a flat surface with the diffractometer axis.

(2) The RTS method gives more homogeneous density than side loading in a powder mount. Generally the bottom part of the powder prepared by side loading is looser than the upper part due to side friction. If the powder is packed with one layer, the density is not homogeneous at all. This defect cannot be eliminated even if the powder is side packed with several layers. Furthermore, based on the authors' experience, it is very difficult to obtain a powder mount with a smooth surface if it is side packed with several layers. When the cover glass is removed, the contact surface between adjacent layers tends to lose some particles, which becomes more severe for loose-packed powder.

(3) As the shape of the cavity in the sample holder is considered, the RTS method has more flexibility than side loading. A rectangular cavity is required by side loading, while the RTS method can pack the powder into a cavity with any shape.

(4) Though particles in a side-loaded powder mount do not orientate parallel to the holder surface, they have significant preferred orientation perpendicular to the side-packing direction. Therefore, reflections from some planes may be of excessively high intensity.

One major disadvantage of the RTS method is that the powder is relatively loose and hence is not very stable if it is placed vertically in a diffractometer with vertical axis. However, with the modified sample holder shown in Figure 1, this problem can be eliminated if care is taken to prevent any shaking or disturbance to the powder mount before examination.

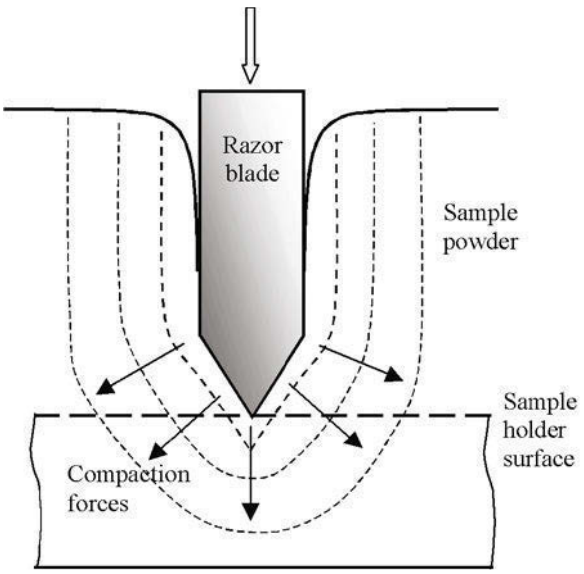

Figure 4. Schematic illustration of compaction directions caused by the sharp blade edge.

CONCLUSIONS

The new RTS method for making random powder mounts for XRD analysis was evaluated and the mechanism by which the method minimizes preferred orientation was analyzed. Comparison between different simple sample-mounting methods indicates that minimum preferred orientation and minimum packing density exist in the powder mount prepared by this method. The results obtained by the RTS method are reproducible. The quartz powder used in this study exhibits a tendency to preferred orientation, indicating that the RTS method can effectively minimize preferred orientation for both platy and anhedral particles.

As pointed out by Brindley (1980), a near-random orientation can be achieved, but strictly random orientation is probably unattainable in practice. It is clear that the RTS method produces less preferred orientation than side loading, even though Brindley (1980) claimed "the side-packed holder is probably the best simple technique to use". Therefore, the RTS method should be considered as a significant improvement toward perfect randomness over side loading, as far as the randomness and other advantages are concerned.

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