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Interactive comment on "Structural Disorder of Graphite and Implications for Graphite Thermometry" *by* Martina Kirilova et al.

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Oohashi-San,

Thank you for spending the time to leave an interactive comment in the open discussion of our manuscript. We acknowledge your expertise in experimental studies on graphite, and thus we are very pleased to receive a positive feedback from you. We consider your comments greatly beneficial for a successful revision of this manuscript, and thus we have taken account of your suggestions to provide more observational data in a revised version of the manuscript. Below we have copy-pasted and then responded to the comments you made in your review.

General comments

Reviewer's comment: (1) The discussion about R2 and "shear strain" The main conclusion of this manuscript is R2 value of graphite increases (which implies decrease of crystallinity) with increasing applied "shear strain". However, "shear strain" you calculated corresponds to "bulk shear strain", and the bulk shear strain and microscopic shear strain which exactly concentrated into the narrow slip zone is quite different. Degree of compaction may differ depends on normal stress applied, hence the "bulk shear strain" reflects not only shearing but also compaction. Also, I would suspect thinning due to leakage of the gouge took place under 25 MPa experiments, especially for slip rate of 100 μm/s (Exp.10). All these issues make it difficult to extract an effect of shearing on the increments of R2 value. To solve this problem, I would suggest using total frictional work (shear stress*displacement) in addition to shear strain, and to discuss its relationship to R2 value.

Response: This issue was also noted by another referee and we have documented our response to both comments in the other response to review. We agree that the measured bulk shear strain is most probably significantly lower than the shear strain accumulated only within the thin shear surfaces. However, we expect the shear strain variations within these surfaces to be linearly correlated with the measured bulk shear strain within a sample, but we recognise and acknowledge that we are only able to calculate a 'rough approximation' from our experimental data. In addition, we have now calculated frictional work as you suggested (Table 2 and Fig. 3b). Reviewer's comment: (2) The relationship between R2 value and graphite "crystallnity" As another referee also mentioned, D bands (and R2 value) reflect amount of grain boundary (edge of grapheme sheet) in addition to intracrystalline defects, so determining which process is dominant in your setting becomes another problem to be addressed.

Response: As we reported in a previous referee's response, we attempted to avoid grain boundaries as much as possible, which was easily done due to the fact that the laser spot size (412 nm) was much smaller than the graphite grains in our samples (>10 microns, fig 4b). We acknowledge that some of the spectra may have been affected by an increase in the grain boundary density, however occasional measurements of this sort are unlikely to affect the average R2 per sample. Thus, we believe that the detected increase in D bands in our experimental data reflects disorder of the internal structure of graphite rather than grain size reduction. Nevertheless, a discussion addressing this topic is added in lines 225-233. Reviewer‘s comment: I could faintly see very small platelets of graphite (< 1μm) in your photograph (Figure 4e), but damages during shin-section making also make this kind of roughness. I think it is better to provide high-resolution SEM images of the slip surface if you could not make good thin section.

Response: (1) We collected Raman spectra directly from the top of the sheared graphite gouges (referred to as shiny surfaces in the manuscript) to avoid damage induced during thin section making (the thin sections were made from other parts of the preserved experimental samples). High resolution SEM images of these surfaces (Fig. 4a and b) were also collected directly from the tops of the layers (labelled as XY sections in the manuscript). (2) We imaged the zone underlying the shiny surfaces by cutting thin sections perpendicular to them. Thus, the small platelets of graphite (< 1μm) in Figure 4e, that you refer to, might have been affected by sample preparation. However, Raman spectra were not obtained from these samples, and thus the reported D bands do not reflect any damage induced during thin section making.

Reviewer‘s comment: On the other hand, I think you should mention that the friction still remains low and stable if you applied shear strain >40. This feature may suggest the graphite on the slip surface still maintain its crystal perfection. In that sense, increments of R2 value of sheared graphite attributable mainly to grain size reduction but not amorphization.

Response: Friction coefficients remain low and stable throughout all experiments. This is reflected in Fig. 1 and in lines 185-186. Our microstructural data clearly indicate partial structural disorder of the graphite structure, so we don’t think that the reduction in the Raman spectra can be reasonably attributed just to grain size reduction. Also, please note there is no evidence of complete amorphization – graphite in all experiments remains crystalline even though significant defects in the graphite structure were introduced.

Specific comments: Reviewer's comment: Line 46; I would happy if you add Oohashi et al. (2013, JGR) in the reference. Response: The citation has been added into the reference list (in lines 378-380). Thank you for reminding us to properly acknowledge this excellent previous study.

Reviewer's comment: Line 60; I think initial thickness of gouge layer varies depends on applied normal stress if you put same weight of graphite powder for each experiments (becomes thinner under high normal stress). Did you change amount of graphite for normal stress of 5 MPa and 25 MPa to ensure to form exactly the same 3-mm thickness? This question is arises from why large shear strain is calculated from the experiments under 25 MPa normal stress (off course, I understand your explanation about compaction). Additionally, I would suspect thinning due to leakage of the gouge took place under 25 MPa experiments, especially for slip rate of 100 μm/s (Exp.10) because the gouge thickness seems to became less than half of the initial thickness. Response: The mass of graphite was not changed throughout the various experiments and hence we acknowledge the effect of normal stresses on the attained shear strain in lines 190-192 (as you have also noted in your comment). Your concern about potential leakage of gouge material during Exp. 10 is based on the dramatic layer thinning recorded at the end of this experiment. However, figure 1b clearly shows a linear trend of increase in shear strain (based on layer thinning) with increase of sliding velocities in the experiments under normal stresses of 25 MPa (ranging from 21,45 through 31.86 to 46,77 in Exp. 8, 9 and 10 respectively). Therefore, we believe that leakage of gouge material is unlikely to have affected the measured layer thickness.

Reviewer's comment: Line 109-110; The authors documented μss does not depend on slip rates, and it remains constant for all experiments. However, I see clear relationship between μ at d=14-20 mm and slip rates; μ decreases with decreasing slip rates for σn=5 MPa, and μ decreases with increasing slip rates for σn=25 MPa. Response:

Dependence of μss on slip rates is not suggested by our data (Table 1). Instead we observe slight variations in 2 of the performed experiments (Exp. 4 and 10). This is reflected in the manuscript in lines 119-120.

Reviewer's comment: Line 141-143; The authors explain graphite crystalinity decreases with increasing slip rates for samples sheared under _n=25 MPa, and no slip rates dependence is found for samples sheared under _n=5 MPa. However, as you concluded, increase of R2 value can be attributed to applied shear strain but not to slip rates. I think you can not discuss direct relationship between R2 value and slip rates unless you conduct various slip-rates experiments at exactly the same shear strain. Response: In lines 151-153, we simply compare the observed decrease in graphite crystallinity with the conditions of the experiments but do not imply direct relationship between R2 and slip rates. Then (lines 153-154), we conclude that 'graphite appears as most disordered in the experiments where the highest shear strain was achieved'.

Reviewer's comment: Line 181; I would suggest referring Di Toro et al. (2011, Nature) instead of Nakatani (2001). Response: Thank you for the suggestion. The reference is now updated. (line 198)

Reviewer's comment: Table 2 and Figure 3 Please add errors and error bars for R2 value. Response: Error estimates are now added in table 2 and line 432.

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