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Subject to modifications and additions.
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Round Robin Project – Results

Shear tests on limestone powder CRM-116 with Ring Shear Testers RST-XS and RST-01.pc

1 Introduction

Early 2008 a round robin project with ring shear testers was initiated. The goal was to determine a range of results for a defined bulk solid, similar to a round robin project with Jenike shear testers carried out in the 1980s [1]. All users of automatic ring shear testers RST-XS or RST-01.pc were invited to take part. 27 labs supplied results. Some labs provided results of multiple tests carried out by different persons, at different ambient conditions, or at different testers. Finally, per stress level up to 60 yield loci (21 labs) measured with the RST-XS, and up to 19 yield loci (10 labs) measured with the RST-01.pc have been received.

In the present document, which should be understood as a preliminary version, the results are subsumed and discussed. The reason for the preliminarity is that possibly further test results may be received in future.

2 Sample preparation and test conditions

The conditions for the tests have been distributed in a document [1]. The most important conditions were:

1. The shear tests had to be done using limestone powder CRM-116, available from the Institute for Reference Materials and Measurements of the European Commission. The material is delivered with “certified” results“ [2], which have been determined with Jenike shear testers in the 1980s.

2. In order to take into account the possible influence of air humidity without making test procedure and preparation too complicated, the following procedure was proposed:

The powder sample should be stored in contact to the lab atmosphere until equilibrium is reached, minimum two days (temperature between 20°C and 25°C, humidity between 30% RH and 50% RH). The lab atmosphere should be stable during this time. Temperature and humidity should be monitored and communicated with the test results. If a humidity chamber is available, it is recommended to use it for the storage of the limestone powder prior to the tests. Temperature and humidity should be adjusted to 20°C and 52% RH (according to proposal in the report of the Jenike Shear Tests [2]).

Participants having the ability to store the powder in a humidity chamber were asked to run the shear tests also with a powder specimen equilibrated at 30% RH and 40% RH (all at 20°C).

3. Yield loci should be measured by all participants at stress levels 1 and 2 (table 2.1), if possible also at stress levels 3 and 4. The stresses in table 2.1 correspond to stresses applied in the Jenike Shear Tester project; just the number of different stresses is reduced.

   In the RST-CONTROL 95 software “N-RHOB-correction” should be activated. In this case RST-CONTROL 95 adjusts the normal load in the shear plane under consideration of the actual bulk density. All other parameters (shear velocity, tolerance, patience) should be set to their default values.

4. For all yield locus tests a standard shear cell should be used (RST-XS: shear cell XS-Mr; RST-01.pc: shear cell M). Use a freshly prepared cell for each yield locus test.

Stress level	normal stress at preshear [Pa]	normal stress at shear to failure [Pa]			
		1st point	2nd point	3rd point	4th point
1	3000	1000	1500	2000	1000
2	6000	2000	3000	4000	2000
3	9000	3000	5000	7000	3000
4	15000	5000	7000	9000	5000

Table 2.1: Stresses for yield locus tests

3 Evaluation

Only those test results have been included in the evaluation which have been determined in accordance to the prescribed test conditions (stresses, shear cell type). For all tests the shear stresses measured at preshear and shear to failure have been calculated using the evaluation software RSV 95. Since some participants did not activate the N-RHOB-correction in RST-CONTROL 95, differences of the normal stresses σ_{meas} to the desired values σ_{target} (table 2.1) of up to 30 Pa for the RST-XS and up to 40 Pa for the RST-01.pc took place. The reason for the latter is that without N-RHOB-correction RST-CONTROL 95 adjusts the normal load so that the desired stress is acting at the underside of the shear cell lid. For the evaluation the normal stress in the shear plane is determined which is somewhat larger due to the mass of the bulk solid between underside of lid and shear plane.

The deviation of the normal stresses from the desired values has been corrected by a procedure like the “prorating” procedure where a new shear stress τ_{new} has been determined from the measured shear stress τ_{meas}:

$$\tau_{\text{new}} = \tau_{\text{meas}} \cdot \frac{\sigma_{\text{target}}}{\sigma_{\text{meas}}} \tag{1}$$

σ_{target} is the normal stress according to table 2.1, σ_{meas} is the normal stress acting in the shear plane.

For every normal stress in table 2.1 a mean shear stress, τ_m, has been determined from the corresponding shear stresses, τ_i, of the individual tests.

$$\tau_m = \frac{1}{n}\sum_{i=1}^{n} \tau_i \tag{2}$$

n: number of measurements

The standard deviation of the shear stresses is the square root of the sample variance:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(\tau_i - \tau_m)^2}{n-1}} \tag{3}$$

The results distributed with limestone powder CRM-116 contain values of the 95% confidence interval for the shear stresses. The confidence interval is calculated based on Student's t-distribution [2]:

$$a = t \cdot \frac{s}{\sqrt{n}} \tag{4}$$

t is Student's t factor which depends on desired probability (here 95%) and number of tests.

Thus, the confidence interval is

$$\tau_m \pm a \quad \text{or} \quad \tau_m \pm t \cdot \frac{s}{\sqrt{n}}.$$

A 95% confidence interval must not be regarded as defining the range of values where one individual test point would lie with 95% probability. With a 95% confidence interval usually a range is defined where an unknown value (here: "true" shear stress), determined by n measurements, lies with a probability of 95%. To find this range, first a mean value of the statistically fluctuating measurement values is calculated according to eq.(2), and subsequently the confidence interval is determined using eq.(4). Then it can be stated that the unknown true value (e.g. the unknown concentration of a mixture) that the true value lies within the confidence interval with 95% probability (The probability is the same for any value within the confidence interval). In analogy to this, concerning the shear tests it could be stated that the true value of the shear stress lies with 95% probability within the 95% confidence interval. However, the latter is only valid for the case that the identical bulk solid is tested, i.e., the moisture and other parameters must be identical (more will follow in section 4). Otherwise, there were no single "true" value.

From equation (4) it follows that a, and thus, the width of the confidence interval, decrease with increasing number of tests, n. This way it is taken into account that with increasing number of tests more information is available, which results in a more accurate statement about the range where the mean value can be found with a certain probability (more accurate = smaller width of the confidence interval).

If the number of tests, n, is greater than 6, a becomes smaller than the standard deviation, s (see eq.(5); for a probability of 95% and $n = 7$ it follows t = 2,447 and a = 0,925s). The greater the number of tests, n, the more individual measurement values (shear stress τ_i) lie outside of the confidence interval.

Since in the frame of the round robin project a large number of test values has been provided (especially for the ring shear tester RST-XS), the 95% confidence intervals of the individual test points are very narrow (see explanation above), i.e. the value a in eq. (4) is much smaller than the standard deviation, s. If in the future someone runs tests with CRM-116, he/she would find that it is nearly impossible to match the narrow confidence interval. As outlined above, the 95% confidence interval does not define a range where future test points would lie with 95% probability.

With the discussion above it is shown that the confidence interval, although it is applied in [2], is not suitable for the assessment of further measurements. Thus, in the following foremost the standard deviation is regarded. For comparison also the standard deviations calculated in {2] for the Jenike shear tester are mentioned.

If a normal distribution of the measured shear stresses is assumed, about 68,3 % of all values lie in the interval $\tau_m \pm s$, and about 95,5 % in the interval $\tau_m \pm 2s$. Since the confidence interval of the mean value is rather small, it can be stated (with some simplification) that the probability of a future measurement value lying within $\tau_m \pm s$ is about 68%, and the probability to lie within $\tau_m \pm 2s$ is about 95%. This statement is not totally exact but allows an assessment of future test results.

Regarding the confidence interval it has to be added that its calculation does not make sense if the humidity of the tested specimens is different due to the influence of the air humidity. In this case one does not measure properties of identical specimens, so that there is no single “true” mean value of the shear stresses.

4 Influence of ambient conditions

An effect of temperature on the limestone’s flow properties is neglectable in the range of typical room temperatures, but the air humidity has a noticeable influence, as already stated in [2]. Therefore, the preparation of the samples and the range of air humidity were prescribed [1]. It would have been ideal if all participants had been able to prepare and test the specimens at the same ambient conditions. To fulfill this, each participant had to run the tests in a climate-controlled lab. However, such equipment was not available for all participants. Thus, in order to get results from as many as possible participants, also results gained at ambient conditions out of the specified range have been used for the evaluation. This led to results from measurements carried out at the following conditions:

- Storage and measurement at the same known conditions
- Storage and measurement at the same, but unknown conditions
- Storage at known conditions, measurement at known, but different conditions
- Storage at known conditions, measurement at different, unknown conditions

Some participants varied the conditions for the sample preparation (storage) in order to provide test results for different conditions. So, the results received cover a range of temperature from 18°C to 25°C and a range of air humidity from 28%rH to 70%rH.

The influence of air humidity on shear stresses was already studied for the tests with the Jenike shear tester [2]. At least in the range above 40% relative humidity (40%rH) an increase of shear stress with increasing air humidity can be noticed. With the ring shear tester RST-XS the author observed a similar increase of shear stress with air humidity. The comparison of tests conducted at 34%rH and 50%rH (storage and measurement at the same conditions) showed that the shear stress increased by about 5 Pa per humidity increase of 1%rH (mean shear stress increase for stress level 1). For the same range of humidity and similar stresses a similar increase of stress was found with the Jenike shear tester [2].

Towards greater normal stresses the absolute increase of shear stress with increasing air humidity becomes larger. At stress level 4 the mean increase is about 10 Pa per 1%rH. Thus, the ratio of shear stress increase per 1%rH to normal stress decreases with increasing normal stress. This is plausible since the influence of adhesive forces, which depend on the amount of water adsorbed on the particle surfaces, is generally smaller at higher stress levels [3]. However, it has to be noted that the data base is too small for an accurate quantitative statement on the influence of air humidity. For this a larger number of tests would be necessary conducted at defined ambient conditions at storage and measurements.

Some of the received results indicate that the material, if first stored under defined conditions, but then tested (including filling of the shear cell etc.) at **different** conditions, may change its properties relatively quickly. Apparently, the adsorption layers adjust to the ambient atmosphere within a short time. Thus, it may play a role how long the container with the test material was open, how quick a shear cell is filled, how large the difference between the air humidity at storage and testing is, and whether a fresh sample from the climate chamber has been used for each test. Since it was not possible to prescribe these procedures in detail and only few participants had the possibility to adjust the ambient conditions during storage and measurement, it is to be expected that during testing the condition of the limestone powder was not always identical to the equilibrium condition during storage.

From the reasons discussed above it may be assumed that the condition of the limestone powder during the test was not in all cases well defined. This must be taken into account at the assessment of the results.

The discussed variation of the ambient conditions should not devaluate the round robin project because ultimately it is a concession to reality. Only few labs are equipped to conduct storage and testing at the same defined conditions. Thus, if the present test results are used to define a range of shear stresses, this range contains the influence of the different ambient conditions. Therefore, if in the future someone will compare its own results with the range obtained here, less effort is required to control the ambient conditions at testing. The price to be paid to for this is that the standard deviation of the results of the present round robin project is larger than it would be for identical ambient conditions at all labs

5 Results

The shear stresses of all tests fulfilling the conditions in section 2, paragraph 4, are plotted vs. normal stress in figs. 5.1 to 5.4 for ring shear tester RST-XS, and in figs. 5.5 to 5.8 for ring shear tester RST-01.pc. The points belonging to one yield locus test, including the preshear point, are connected with straight lines. This is **not** the course of the yield locus but shall just show which points are part of one yield locus test. One can see that the lines of the different yield locus tests are mostly parallel to each other. This means that the range of scatter of all tests is not a result of the scatter of individual points of a yield locus but results from the different shear stress levels of individual yield loci.

Please note that the diagrams contain results obtained at different ambient conditions (temperature 18°C to 25°C, air humidity 28%rH to 70%rH). Especially air humidity has a significant influence on the results which will be discussed below.

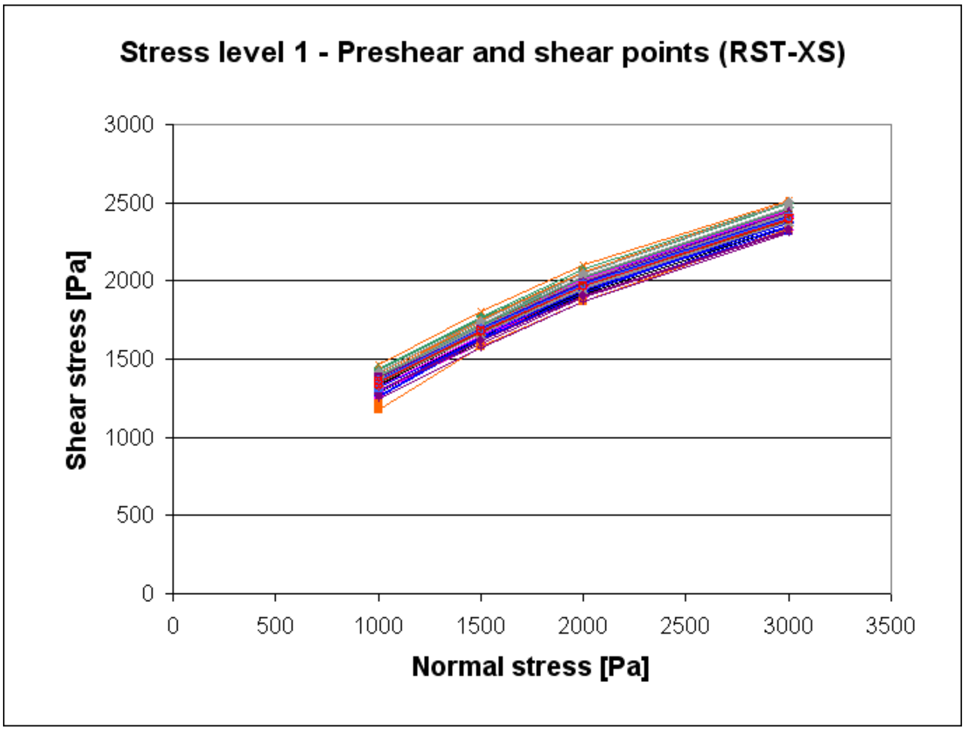

Fig. 5.1: Test points at stress level 1 (RST-XS, 60 yield locus tests)

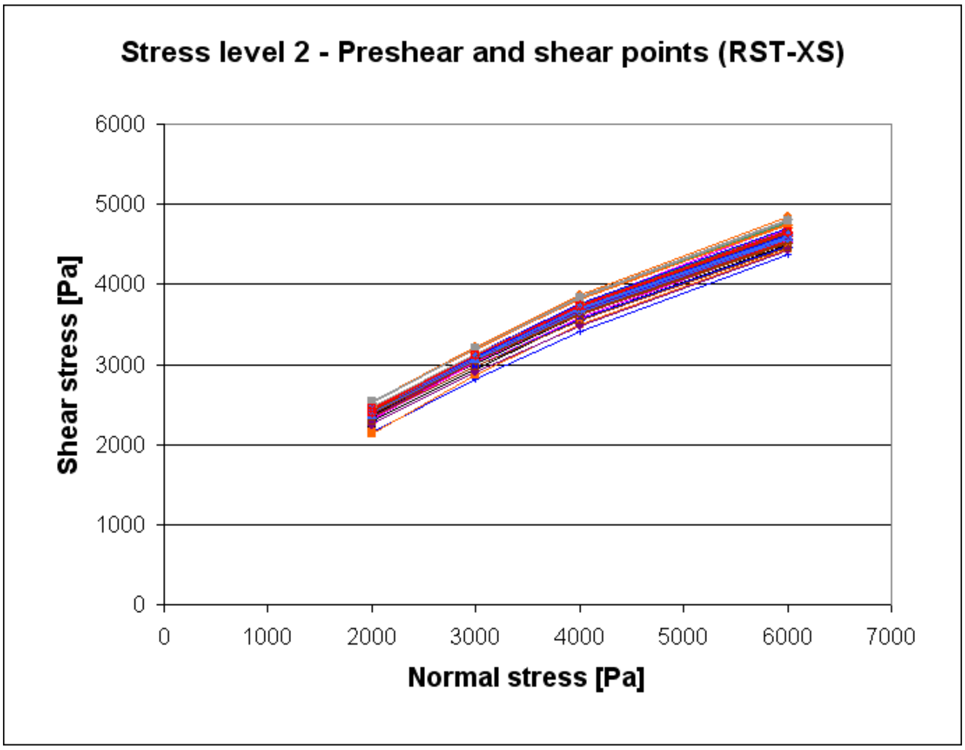

Fig. 5.2: Test points at stress level 2 (RST-XS, 59 yield locus tests)

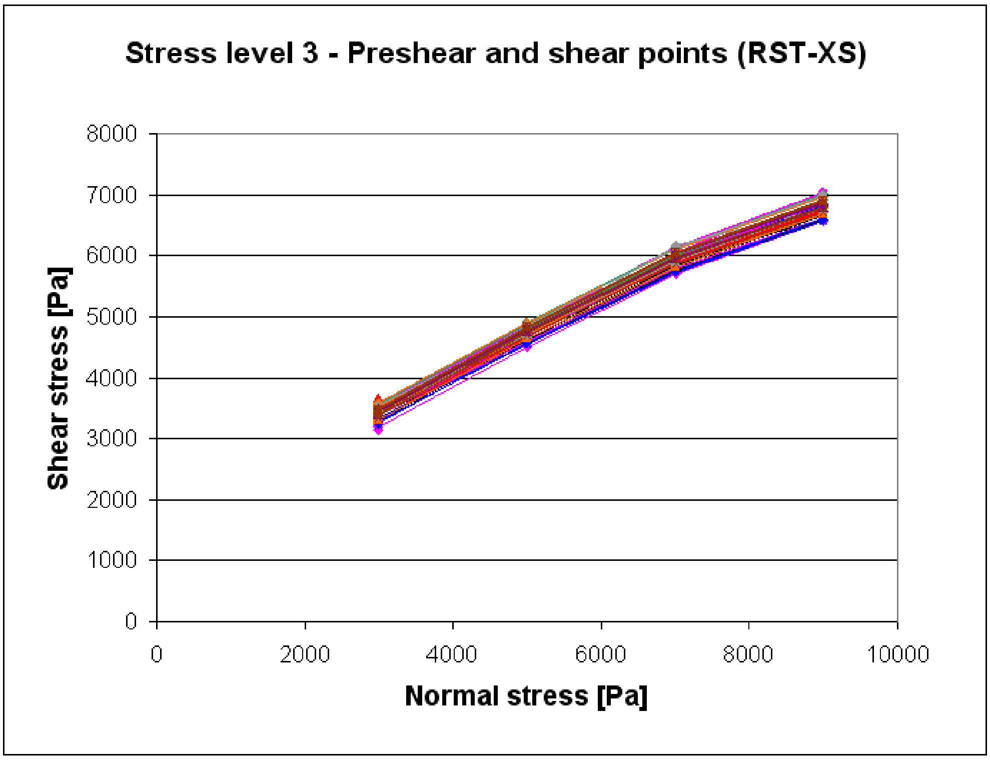

Fig. 5.3: Test points at stress level 3 (RST-XS, 52 yield locus tests)

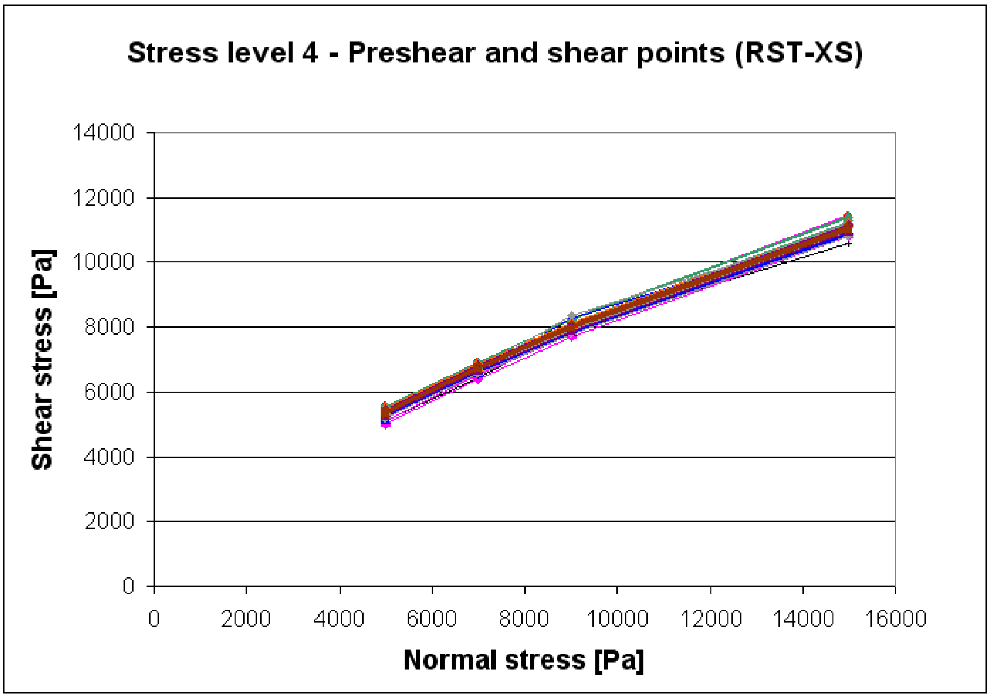

Fig. 5.4: Test points at stress level 4 (RST-XS, 52 yield locus tests)

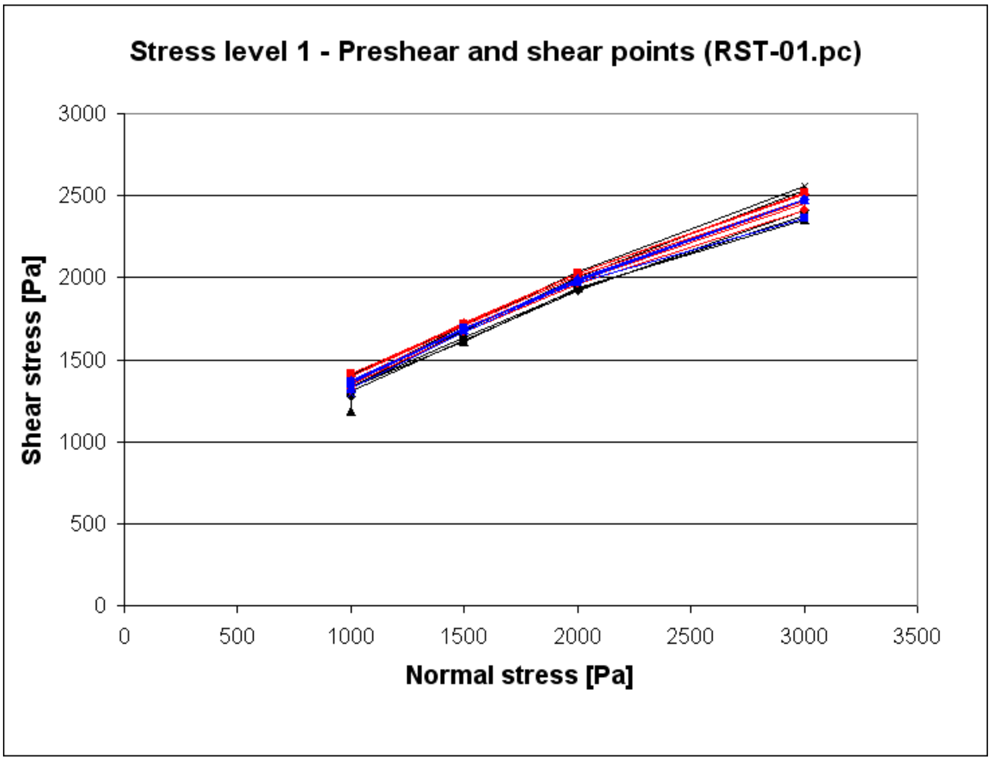

Fig. 5.5: Test points at stress level 1 (RST-01.pc, 19 yield locus tests)

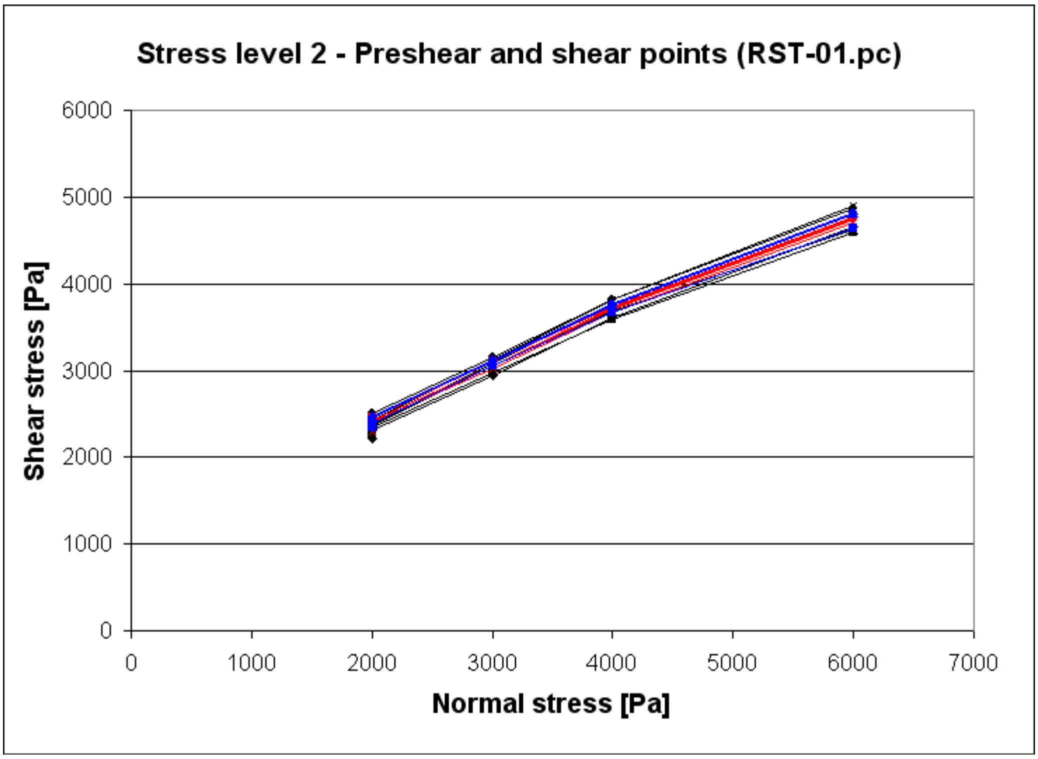

Fig. 5.6: Test points at stress level 2 (RST-01.pc, 17 yield locus tests)

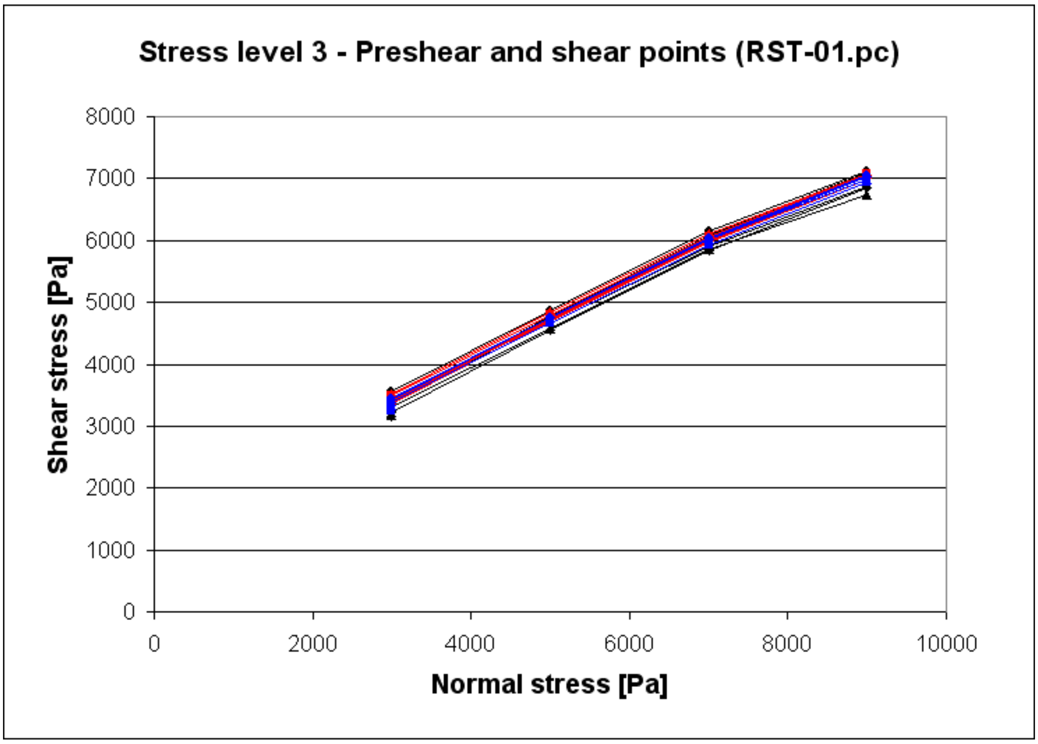

Fig. 5.7: Test points at stress level 3 (RST-01.pc, 16 yield locus tests)

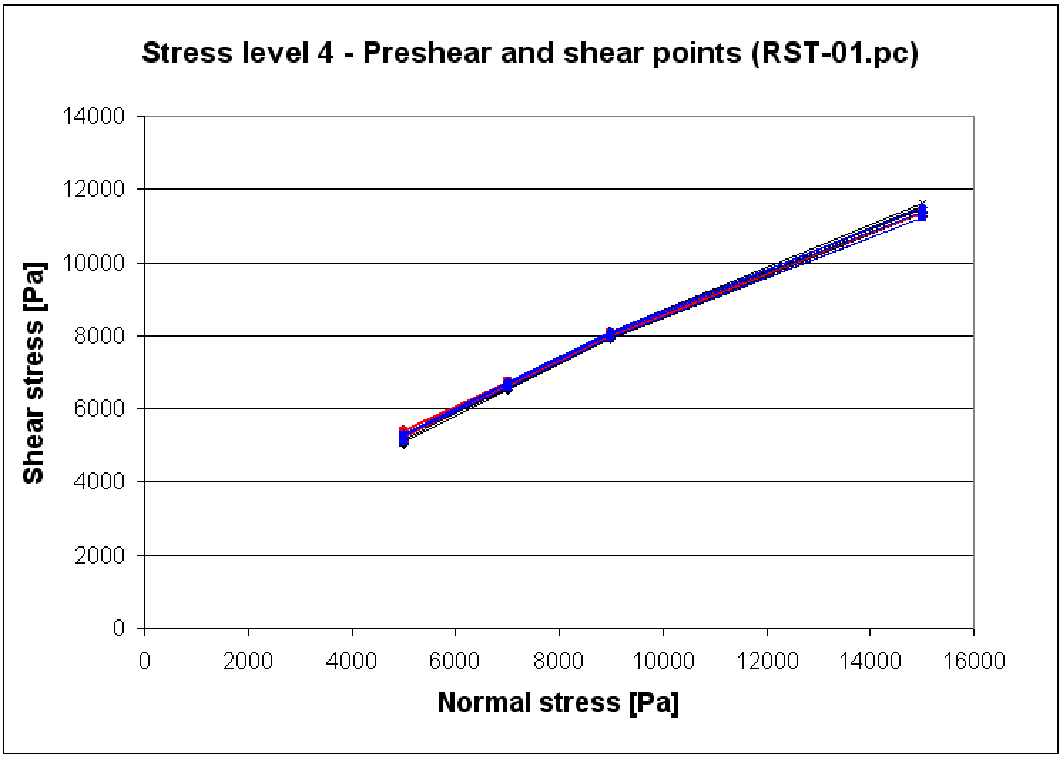

Fig. 5.8: Test points at stress level 4 (RST-01.pc, 16 yield locus tests)

As outlined in section 3, from the tests results both standard deviations and confidence intervals have been calculated in analogy to the evaluations of the Jenike shear tester results in [2]. In fig. 5.9 these values measured with ring shear tester RST-XS at stress level 2 and the corresponding values of the Jenike shear tester [2] are plotted. The diagram leads to the following statements, which are valid also for the other stress levels:

1. Compared to the Jenike shear tester, the ring shear tester provides a clearly narrower range of scatter, which is characterized by the smaller standard deviation and, thus, the narrower interval $\tau_m \pm s$.
2. The shear stresses obtained with the ring shear tester are located in the upper part of the 95% confidence interval $\tau_m \pm a$, or interval $\tau_m \pm s$, respectively, of the Jenike shear tester. For small stress levels, the upper limit of the confidence interval of the Jenike tester is slightly exceeded by the upper limits of the corresponding intervals of the ring shear tester.
3. As discussed above, the 95% confidence interval $\tau_m \pm a$ of the ring shear tester is very narrow (the light red limits of the confidence interval are very close to the mean), which is a result not only of smaller standard deviation s, but also of the larger number of tests, n (see eq.(4)).

Since the 95% confidence intervals of the ring shear testers are very narrow, and according to the discussion in section 3, the confidence interval will not be regarded further in this report. Instead the standard deviation will be applied as a measure for the range of shear stresses.

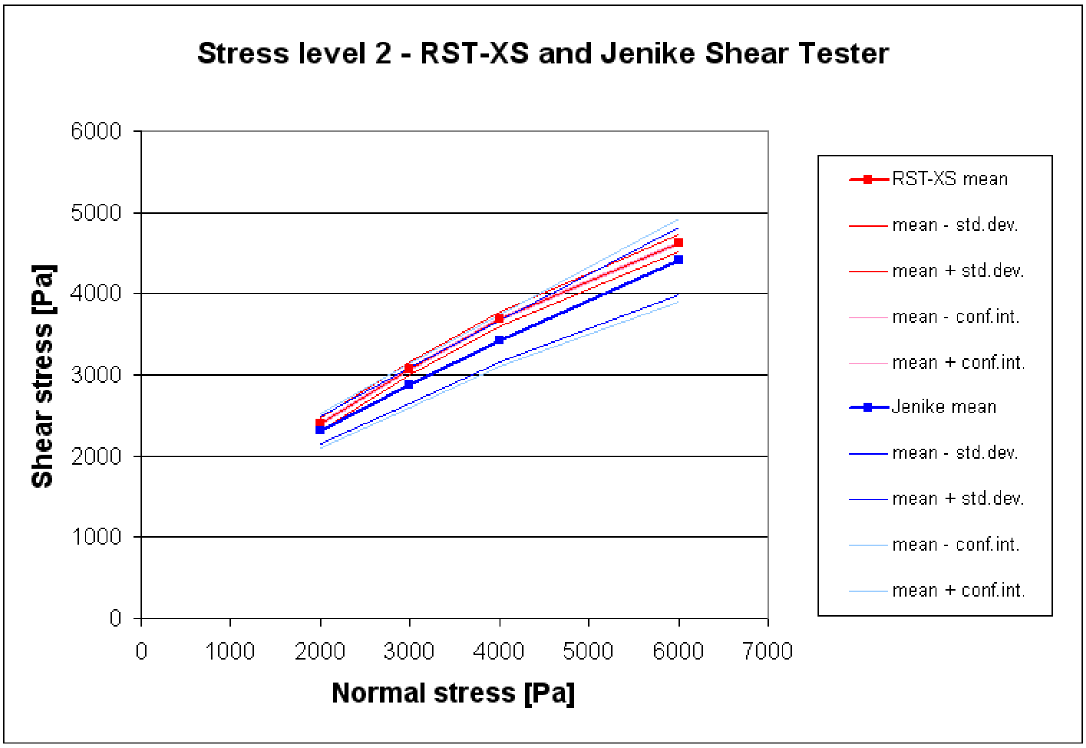

Abb. 5.9: Mean shear stress τ_m (mean), range of standard deviation $\tau_m \pm s$ (mean ± std.dev.) and 95% confidence interval $\tau_m \pm a$ (mean ± conf.int.) of Jenike shear tester [2] (blue), ring shear tester RST-XS (red). Values have been measured at stress level 2.

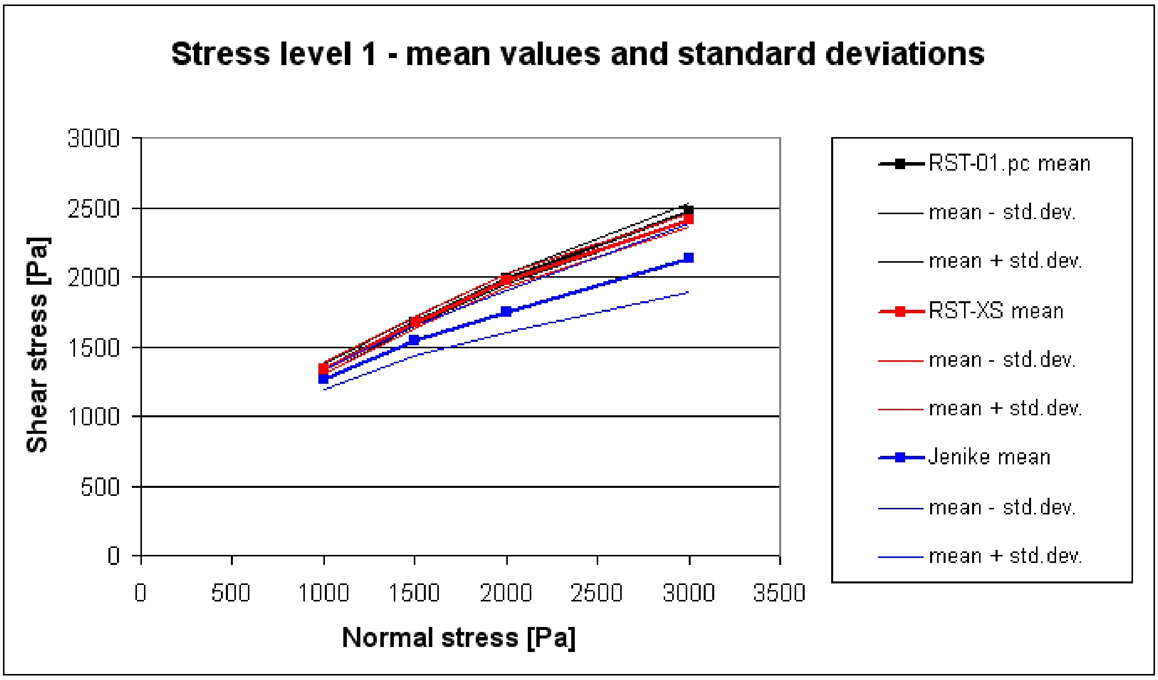

Fig. 5.10: Mean shear stress τ_m (mean) and ranges $\tau_m \pm s$ of standard deviation around mean (mean ± std.dev.) of ring shear testers RST-01.pc (black) and RST-XS (red) and Jenike shear tester [2] (blue) for stress level 1

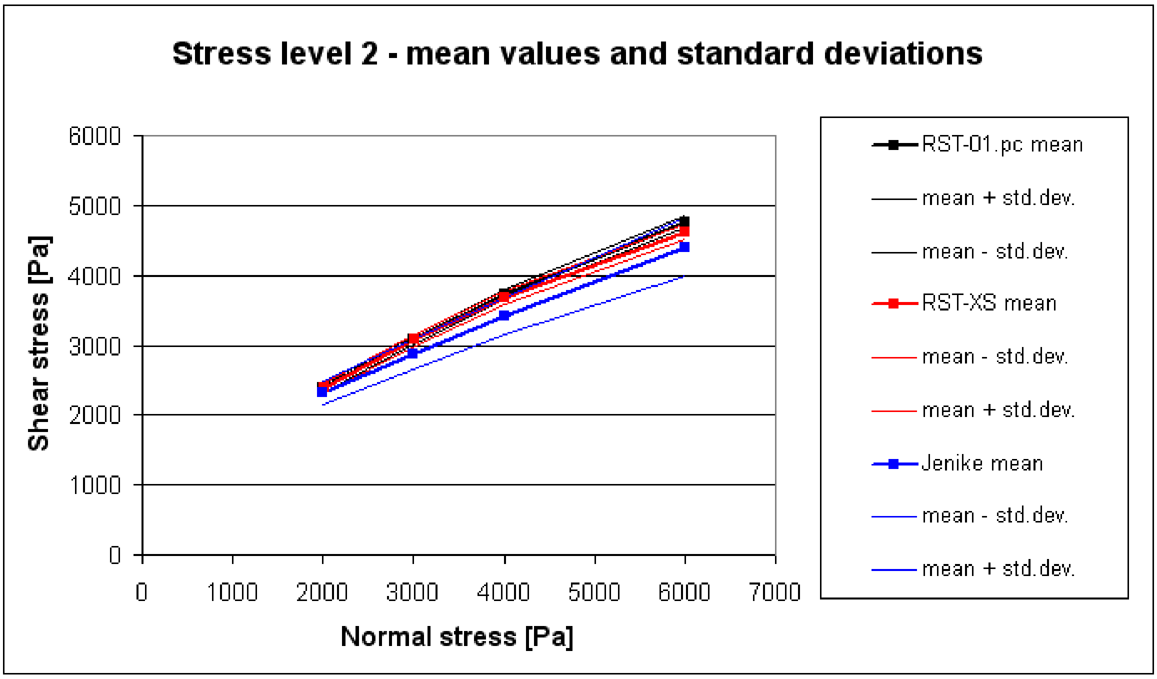

Fig. 5.11: Mean shear stress τ_m (mean) and ranges $\tau_m \pm s$ of standard deviation around mean (mean ± std.dev.) of ring shear testers RST-01.pc (black) and RST-XS (red) and Jenike shear tester [2] (blue) for stress level 2

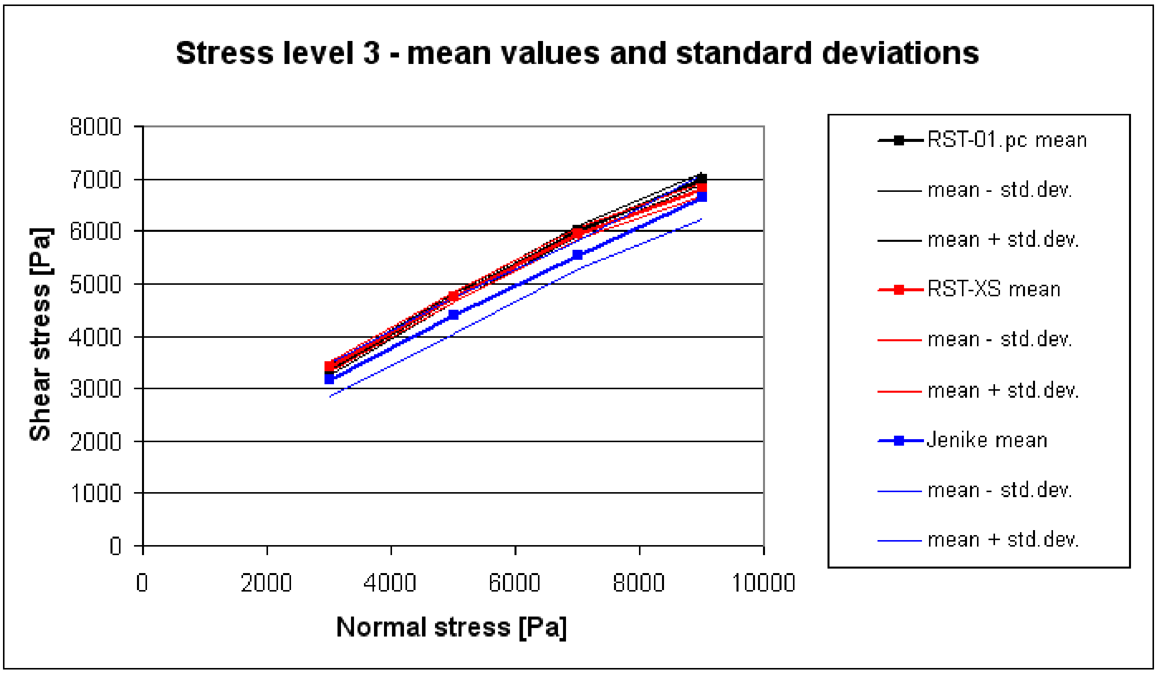

Fig. 5.12: Mean shear stress τ_m (mean) and ranges $\tau_m \pm s$ of standard deviation around mean (mean ± std.dev.) of ring shear testers RST-01.pc (black) and RST-XS (red) and Jenike shear tester [2] (blue) for stress level 3

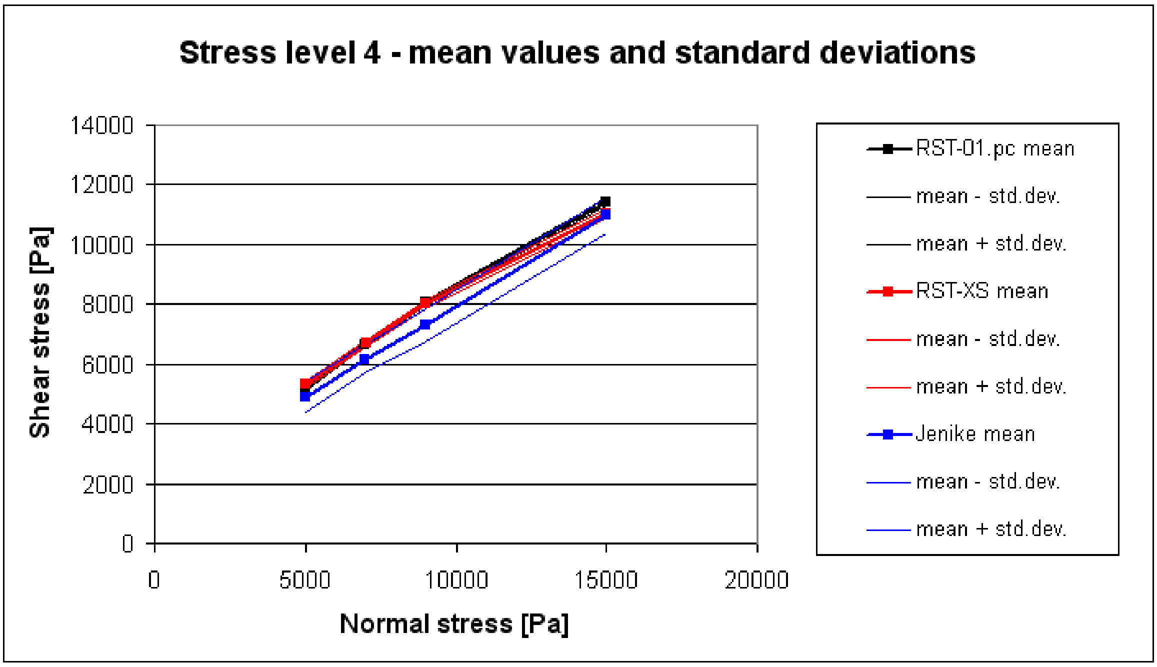

Fig. 5.13: Mean shear stress τ_m (mean) and ranges $\tau_m \pm s$ of standard deviation around mean (mean ± std.dev.) of ring shear testers RST-01.pc (black) and RST-XS (red) and Jenike shear tester [2] (blue) for stress level 4

Stress level	σ [Pa]	RST-01.pc		RST-XS		Jenike Shear Tester [2]	
		τ_m [Pa]	s [Pa]	τ_m [Pa]	s [Pa]	τ_m [Pa]	s [Pa]
1	1000	1337	41	1342	47	1268	75
	1500	1680	31	1678	45	1540	108
	2000	1990	35	1976	49	1749	149
	3000	2471	63	2412	52	2138	245
2	2000	2381	66	2394	78	2318	166
	3000	3078	56	3076	81	2873	219
	4000	3727	65	3686	90	3419	256
	6000	4768	93	4624	101	4409	414
3	3000	3359	94	3414	92	3156	319
	5000	4729	85	4741	97	4385	345
	7000	6008	87	5951	113	5546	259
	9000	7000	105	6801	126	6655	419
4	5000	5230	104	5321	111	4905	517
	7000	6670	71	6700	114	6155	464
	9000	8043	75	7995	137	7308	538
	15000	11420	108	11053	166	10992	615

Table 5.1: Mean shear stress τ_m and standard deviation s of results from ring shear testers RST 01.pc and RST-XS (temperatures 18°C to 25°C, air humidity 28%rH to 70%rH) and Jenike shear tester [2] for all stress levels

In figs. 5.10 to 5.13 the mean shear stresses τ_m with the ranges $\tau_m \pm s$ (mean ± std.dev.) are plotted for ring shear testers RST-XS and RST-01.pc. In addition, the corresponding values of the Jenike shear tester taken from [2] are plotted. The numeric values are listed in table 5.1.

The results show that the shear stresses of both ring shear testers RST-01.pc and RST-XS are close to each other. Also, the standard deviations are similar.

The ratio of the standard deviation to the mean shear stress (s/τ_m) is shown in table 5.2. The ratios are similar for both ring shear testers, mostly between 1.5 % and 3 % (including the influence of the different air humidity). For the Jenike shear tester values in the range of about 5 % to 11 % have been found. The ratio s/τ_m of the ring shear tester results decreases with increasing stress level which is also visible in figs. 5.10 to 5.13 where with increasing stress level the range $\tau_m \pm s$ becomes narrower in relation to the measured shear stresses. The most important reasons for this behaviour may be:

1. The relative increase of shear stress with increasing air humidity is smaller at higher stress levels (section 4).
2. A possible influence of the bulk solid's stress history (e.g., filling) will be less severe at high stress levels. The reason for this is that shearing a bulk solid at high stresses changes its structure more than shearing at lower stresses. This was observed on shear tests at very small normal stresses [4].

Stress level	σ [Pa]	RST-01.pc	RST-XS	Jenike Shear Tester [2]
		s/τ_m [%]	s/τ_m [%]	s/τ_m [%]
1	1000	3.06	3.48	5.89
	1500	1.84	2.69	7.01
	2000	1.76	2.48	8.50
	3000	2.53	2.14	11.47
2	2000	2.76	3.26	7.18
	3000	1.83	2.62	7.62
	4000	1.75	2.43	7.49
	6000	1.96	2.18	9.38
3	3000	2.81	2.70	10.10
	5000	1.80	2.05	7.88
	7000	1.45	1.90	4.67
	9000	1.50	1.85	6.29
4	5000	2.00	2.09	10.53
	7000	1.06	1.70	7.53
	9000	0.93	1.72	7.36
	15000	0.95	1.50	5.59

Table 5.2: Ratio of standard deviation, s, to mean shear stress, τ_m, of results from ring shear testers RST-01.pc and RST-XS (temperatures 18°C to 25°C, air humidity 28%rH to 70%rH) and Jenike shear testers [2].

To assess the influence of air humidity, tests performed at two different ambient conditions are regarded. At both tests temperature and air humidity during storage (preparation) and measurement were identical. This way a possible change of the powder's properties during shear cell filling and testing can be excluded. Two yield loci have been measured at both conditions 20°C/50%rH and 22°C/34%rH, i.e., at nearly identical temperature, but different air humidity. The measured shear stresses at stress level 1 are shown in fig. 5.14. For comparison also the range $\tau_m \pm s$ is plotted. It is obvious that different air humidity leads to a significant increase of shear stress. A closer examination of the test results leads to the dependence of shear stress on air humidity already mentioned in section 4, which has been detected also with the Jenike shear tester [2].

The difference between the shear stresses measured at different humidity is slightly smaller than the width of the interval $\tau_m \pm s$ (fig. 5.14). Also, at the other stress levels the influence of air humidity is visible. In the mean the increase of shear stress in the humidity range from 34%rH to 50%rH is about 1.0 to 1.5 times the standard deviation, s, of the corresponding shear stresses. Since at some tests the air humidity was out of the range 34%rH to 50%rH, it is likely that for all tests the influence of air humidity on the measured shear stress is larger than that documented fort the range 34%rH to 50%rH. This leads to the conclusion that the standard deviations of the test results of the present study are to a significant extent the result of different ambient conditions, especially different air humidity. In the ideal case a round robin had to be conducted at identical ambient conditions in all labs, both during storage/preparation **and** testing. But, as already discussed in section 4, this had not been possible to realize by all participants, or the effort to achieve this would have been too large.

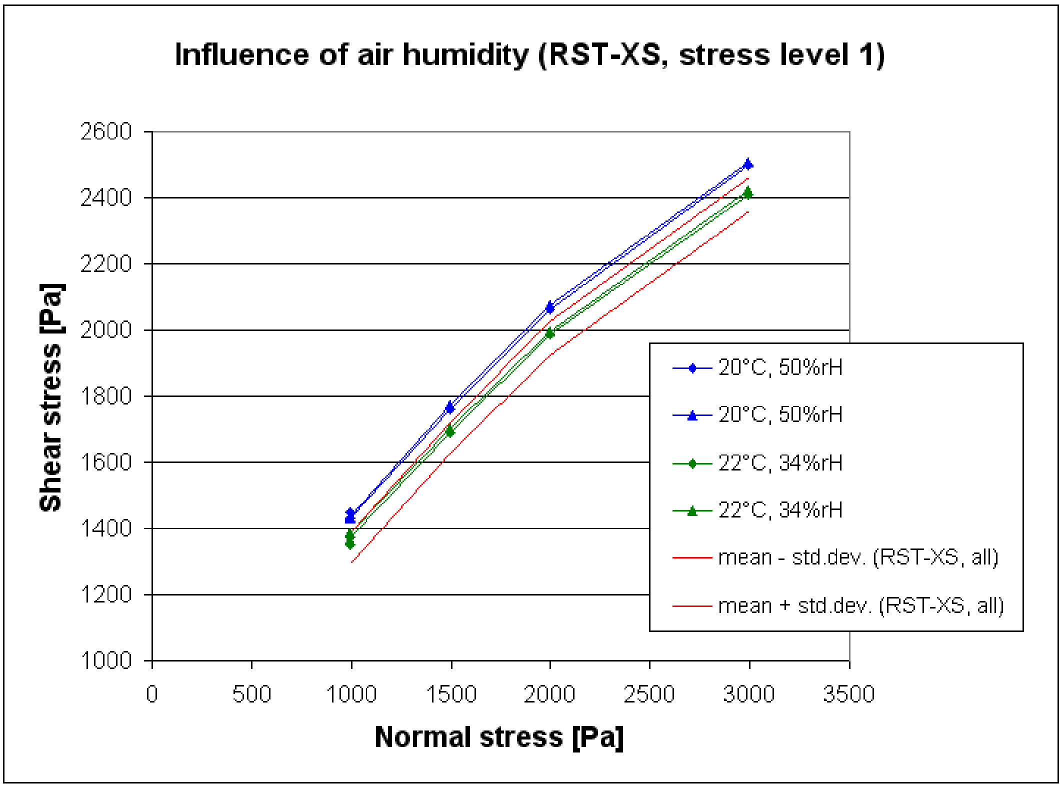

Fig. 5.14: Shear stresses measured at two different ambient conditions; range $\tau_m \pm s$ (mean ± standard deviation) of all RST-XS test results (red curves) for stress level 1

Regarding all received test results, the shear stresses are within a relatively small range although the ambient conditions were different. This is demonstrated by the standard deviations, which are relatively small especially compared to the results of the Jenike shear tester [2].

If only results from one lab measured within a relatively narrow range of ambient conditions are regarded, clearly smaller deviations are possible compared to the standard deviation of all tests. As an example, in fig. 5.15 results of the lab having provided the largest number of tests are shown (RST-XS, stress level 1). Although the limestone powder has been equilibrated at 40%rH and 52% rH prior to the tests, the results are close to each other and lie clearly within the range $\tau_m \pm s$

of all test results. Probably the influence of the air humidity during storage is not clearly visible because the ambient conditions in the lab were the same for all tests and the powder adjusted to these conditions relatively quickly (see above).

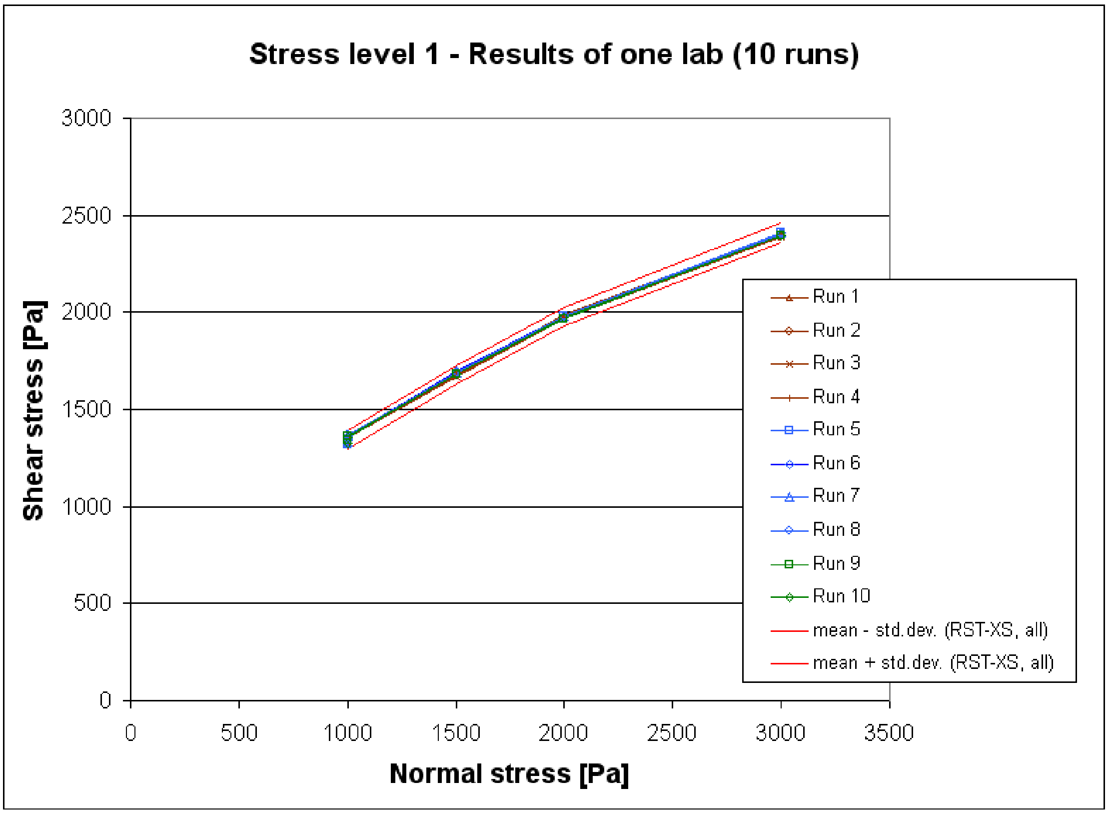

Fig. 5.15: Shear stresses measured by the lab having provided the largest number of test runs (storage of the limestone powder at 40%rH for run 1 to 5 and at 52% rH for run 6 to 10); range $\tau_m \pm s$ (mean ± standard deviation) of all RST-XS test results (red curves) for stress level 1

6 Application of results and further investigations

It was the most important goal of this round robin to define a range of results for a reference powder in a similar way as it has been done with the Jenike shear tester [2]. With such a range, users can check if their tester is able to produce test results matching the reference values. From the view of the author, this is to be expected if a ring shear tester is well adjusted and calibrated, but with the reference powder the opportunity for an additional check is provided.

In table 5.1 mean shear stresses, τ_m, and standard deviations, s, are given. If a normal distribution is assumed, 68.3 % of the test results lie within the range $\tau_m \pm s$, and 95.5 % lie within the range $\tau_m \pm 2s$. To compare own results with this data, one should consider these ranges, i.e., the own results should lie within $\tau_m \pm s$ with about 68% probability, and within $\tau_m \pm 2s$ with about 95% probability (see section 3). Since air humidity has a significant influence on the shear stress and obviously is responsible for an essential part of the standard deviation, the ambient conditions at storage/preparation **and** testing should not deviate too much from the conditions [1] (see section 2).

For the interpretation of the results it would be a big step forward to start a systematic examination of the influence of air humidity. For this, multiple tests with the same defined conditions at storage/preparation **and** testing had to be conducted. Furthermore, it would be interesting to get more information on the adsorption kinetics (how long need adsorption layers to adjust to the ambient conditions?).

7 Summary

In 27 labs in Europe and the United States different persons ran tests with ring shear testers RST-XS and RST-01.pc on a standard powder which has been used in the past for the Jenike shear tester [2]. Despite different ambient conditions (temperature range 18°C to 25°C, air humidity 28%rH to 70%rH) the range of results is relatively narrow, especially at higher stresses. First investigations on the role of the air humidity indicate that with identical ambient conditions during storage, preparation and testing even better agreement of results could have been achieved, because the influence of air humidity on shear stress is in the same order of magnitude as the observed standard deviation.

With the calculated mean values and standard deviations, a reference range can be defined. This range should be matched with a certain probability (see section 6) with the results of future tests with the standard powder if the instructions regarding stresses and ambient conditions are followed. Thus, the results provide orientation to all users of ring shear testers RST-XS and RST-01.pc.

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9 Literature

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