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Nanoindentation in teeth: the influence of experimental conditions on local mechanical properties

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Abstract

The influence of various experimental conditions on the elastic modulus and hardness of human intertubular dentin was studied using instrumented nanoindentation at room temperature. The conditions studied were: dry (chemically dehydrated) versus wet (prepared and nanoindented under HBSS), the influence of long-term storage under HBSS at ca. 4°C, and the influence of deep-freezing under dry and wet conditions, respectively. The reversibility of chemical dehydration and the consequences of multiple deep-freezing cycles were also investigated. Two premolars of a twelve-years-old human male were chosen for evaluation. The absolute values of the mechanical properties differed by a factor of ~ 2 and ~ 3 between dry and wet state, whereas trends were conserved. Deep-freezing wet dentin samples deteriorated their mechanical properties by about 20 to 28%, whereas dehydrated samples suffered no damage. This may be due to the mechanical damage by the volume increase of water freezing inside the tubules.

Keywords: nanoindentation, biological materials, intertubular dentin, experimental conditions, Hank's balanced salt solution (HBSS).

1. INTRODUCTION

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5 Storing teeth is an important matter concerning dental medicine. Keeping the
6 integrity of these materials for further transplantation, replantation and
7 restoration is an important issue for successful dental treatments [1, 2] In
8 addition, in the case of research investigations, it is a crucial factor in order to
9 measure the real mechanical response of such materials in a state as close to
10 in-vivo as possible. Biological samples not studied immediately after harvesting,
11 are usually stored in the laboratories until testing. Several storage solutions
12 have been reported (HBSS; CaCl₂ solutions, water, etc) in order to simulate in
13 vivo conditions and to try to keep the integrity of the teeth. However, these
14 solutions can strongly influence the native properties of these systems [3, 4,
15 etc]
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20 Nanoindentation is a technique able to detect the above deviations in the local
21 mechanical properties of these materials, as Habelitz et al. [5] also
22 demonstrated with three different storage solutions. Although the penetration
23 depth of nanoindentation is small (< 1 μm); the mechanical properties thus
24 obtained are assumed to be representative of the whole sample and are
25 comparable to one of the most accurate ways of determining elastic constants
26 of a material, i.e. sonic measurements [6]. Nanoindentation is a versatile,
27 powerful and highly suitable technique for testing biomaterials, mainly because
28 there is no need of vacuum condition and near in vivo condition can be attained
29 quite easily.
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34 Dentin is a mineralized tissue, containing approximately 50 % vol. mineral and
35 30 % vol. of collagen fibrils. Dentin contains many tubules typically radiating
36 outwards from the root cavity, in which the dentinal fluid is located and the
37 cytoplasmic processes of the cells that have formed the dentin, the
38 odontoblasts, take place. In addition, tubules are surrounded by a thin, hard,
39 highly mineralized shell of peritubular dentin, the contribution of which to the
40 overall mechanical properties of a tooth is not yet quite clear [7 - 9]. Local
41 mechanical properties of the intertubular dentin (between the tubules)
42 investigated here depend on mineral content and level of interpeptide hydrogen
43 bonding. Most storage solutions are known to alter both characteristics [5, 10].
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48 The present study investigates the difference in hardness and elastic stiffness
49 between dehydrated samples and samples prepared and indented under HBSS.
50 Furthermore the influence of the temperature of storage on the measured local
51 mechanical properties is studied, since Panigh et al. [11] found an influence,
52 albeit for the case of bond strength. The reversibility of chemical dehydration is
53 tested, carrying out nanoindentations in the same sample first in wet and then
54 in dried plus rewetted conditions. In addition the suitability of HBSS for long-
55 term storing of teeth is studied by testing one premolar of the same human
56 individual immediately after extraction, and another one after two years of
57 storage in HBSS (plus Na-azid).
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The method of nanoindentation is carried out on intertubular dentin after
storing the corresponding samples from the same tooth under four different

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3 conditions: immersed in HBSS and room temperature, chemically dehydrated
4 and room temperature, immersed in HBSS and deep frozen, and chemically
5 dehydrated and deep frozen. Correspondingly, nanoindentations are done
6 under atmospheric and fluid environments as appropriate.
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9 10 **2. SAMPLE PREPARATION**

11 Preliminary experiments with a human tooth of unknown origin extracted at
12 hospital ended with a thick, very soft surface layer of unknown composition in
13 spite of the use of antibacterial agent and had to be discarded. Another
14 investigated tooth had to be discarded because it had been cut in halves during
15 extraction, which presumably lead to damage due to overheating. These facts
16 prove that in order to get reliable values of mechanical properties of teeth it is
17 of utmost importance to use healthy, freshly erupted specimens, extracted by a
18 dental surgeon fully aware of the requirements of the experiments and thus
19 avoiding pre-damage.
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24 For the investigation reported here two second healthy premolars extracted
25 from a 12 years old human male were examined. Each of them was extracted
26 from each side of the jaw as part of an orthodontical treatment due to a lack of
27 alveolar arch space distal to the second molar.
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30 One of two premolars was mechanically tested immediately after extraction
31 (1P) and the second one (2P) was studied after 2 years of storage immersed in
32 HBSS and kept at $\sim 4^{\circ}\text{C}$.
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35 The samples were cut with a diamond saw in two (1P) and four sections (2P)
36 along the crown-root direction, carefully avoiding any overheating. Half of the
37 samples of the same premolar were immediately dehydrated ('DEH'), soaking it
38 in different ethanol grading; the remainders were immersed in HBSS ('HBSS').
39 In addition, the influence of two temperatures of storage were studied, in the
40 case of 2P samples: room temperature and -15°C (deep freezing), in
41 combination with the 'DEH' and 'HBSS' condition described above.
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45 After the indentation of the (2P) samples treated according to the first
46 experimental conditions, the former dehydrated samples were rewetted (RW) in
47 HBSS and stored at the same temperature as at the beginning (RT or -15°C)
48 and the former HBSS samples were dehydrated ('DEH') in ethanol and stored at
49 the same temperature as in the beginning. The later experiments were done to
50 study the reversibility of chemical dehydration. In addition, one ('HBSS') sample
51 was frozen and thawed 5 times (5x) to investigate the occurrence or not of
52 additional damage with multiple freezing, on the local mechanical properties
53 measured at room temperature.
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57 For better understanding, all the conditions tested, including the nomenclature
58 used, are summarized in table 1.
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'[Insert table 1 about here]'

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For testing and handling purposes, some of the pieces were simply glued to a platform with cyanacrylate glue and some of them were embedded in a polymer resin. In all the cases, the transversal section of the premolar consisting of enamel, dentin and pulp, was accessible for preparation. The exposed area was ground and polished to a 2500 P grit finish with silicon carbide cloths, followed by polishing in 0.3 μm alumina suspension. In the case of 'wet' conditions, the samples were permanently rinsed with HBSS while polishing, and stored under HBSS all the time to avoid accidental dehydration. This is essential especially in the last stages of preparation.

3. NANOINDENTATION TESTS

Nanoindentation tests were carried out using an add-on nanoindentation device (Hysitron Triboscope, Hysitron Inc., Minneapolis, MN, USA) mounted on the scanner head of an AFM stage (Veeco – Digital Instruments, Santa Barbara, CA, USA).

Two kinds of configurations were used in the nanoindenter: on the one hand, 'dry indentations', in which the dehydrated samples were indented under atmospheric conditions with a cube-corner indenter, on the other hand, 'wet indentations', in which sample and part of the indenter were immersed in HBSS during testing. For the latter case the conical indenter tip is mounted at the end of a tungsten rod ca. 9.5 mm long in order to keep the transducer well away from the fluid cell. Unfortunately, it turned out that the long rod sometimes introduced a markedly higher level of noise into the measurements; therefore we preferred the standard tip with the standard short rod for the "dry" measurements. No remarkable influence, on the other hand, was found of the fluid itself on the results. So, all the 'wet' indentations were done with the same conical indenter on a long tungsten rod, whereas all the 'dry' indentations were done with the same cube corner indenter. This procedure is justified by a preliminary investigation [12] showing that the shape of the indenter does not influence the results of H and E in dentin (results not shown here).

Nanoindentation tests were carried out in several loading-unloading steps, the first three steps were the same for (1P) and (2P) samples: first the specimen was linearly loaded to the maximum load in 5 seconds, secondly the load was maintained 50 seconds in order to eliminate most of the creep before unloading, thirdly the sample was unloaded to an intermediate load. In the case of 1P specimens and wet condition, after the third step, 5 cycles of unloading and loading between 10 % of maximum load and maximum load were included and for each indentation, 5 elastic moduli were calculated from the unloading curves and averaged. Hardness was calculated from the first cycle. Since measurements from 1P samples showed no significant difference between E calculated from the five unloading curves, these cycles were not applied to the 2P samples. Regarding (2P) samples, the third unloading step was done until an intermediate load of 100 μN within 10 s, fourth step was to hold this load 20 s for control of thermal drift and, finally, unloading linearly to zero within 2 s.

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Maximum load was 5000 μN in all the samples and conditions, except in the case of the 'dry' indentations of 2P samples, where maximum load was 2500 μN , in order to keep the penetration depth between 700 and 800 nm.

The temperature of testing was in all the cases room temperature. The samples stored in a deep-frozen (-15°C) condition were defrosted before testing.

In the case of 1P specimens, the indentations were done along a line starting in the dentin-enamel junction (DEJ) and finishing in the root, far away from the pulp. On the other hand, in the case of 2P samples, the indentations were done randomly in the dentin zone, far away from the pulp and enamel. In the later, the tests were repeated daily during two weeks in order to check the stability of the measured mechanical properties with time. In both cases, the indented region was intertubular dentin, and the indents were carefully placed by means of the AFM mode scanning with the nanoindenter tip between the tubules to avoid any influence of the peritubular dentin and/or tubules on the results.

Elastic Modulus and Hardness were calculated by means of the load-displacements curves using the well known Oliver-Pharr method [13].

At least 30 measurements per storage condition were taken. In order to compare the results, ANOVA one way statistical analysis was used, with a confidence interval of 0.05.

4. RESULTS

The ratio between the measured mechanical properties, E and H, in the wet and dry state is ~ 2 and ~ 3 , respectively, as can be seen from figure 1a and figure 1b. However, although the absolute values of E and H differ, the trends, such as the typical gradient in E and H starting at the dentin-enamel-junction (DEJ) and extending for some hundreds of micrometers into the dentin are conserved, as shown in figure 2a and figure 2b. In addition a few indentations were made in the enamel, showing that even in this very highly mineralized material (up to 95 wt%, [14, 15]) a strong difference in the hardness exists between the dry and the wet state ($\approx 2:1$), whereas, strange enough, the elastic modulus remains unaltered. But since enamel was not the main aim of this investigation, this was not pursued further.

'[Insert figure 1a and figure 2b about here]'

'[Insert figure 2a and figure 2b about here]'

The measured mechanical properties in the case of 2P samples for each condition were significantly similar during the two weeks of testing. For this reason, all the presented results correspond to the average of the measurements (among 30 to 180 nanoindentations per condition) during the evaluated time.

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5 The final median (average) data of all the initial conditions are shown in table
6 2, followed by their standard deviation.
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8 '[Insert table 2 about here]'

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10 A result to point out is the similarity in the mechanical response of the 1P and
11 2P samples (denoted by dark and light grey cells for the corresponding wet and
12 dry properties, respectively, in table 2), where the main difference is the
13 storage time (immediately tested and stored during 2 years in HBSS,
14 respectively).
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18 As shown in table 2, for the 2P samples, the measured mechanical properties
19 vary with the temperature of storage by 25 %, approx. in the case of wet
20 conditions. This result was not observed in the dry conditions. From now, dry
21 conditions will be denoted by simply 'DEH' (without specifying temperatures of
22 storage) since E and H are not influenced by the temperature of storage in the
23 dry state.
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27 Figure 3a and figure 3b show all the results of the wet and rewetted conditions
28 of 2P samples. Independently of the storage conditions in the dehydrated state,
29 after rewetting, their mechanical properties fit in with the mechanical properties
30 of HBSS RT condition. A similar behaviour is found in the case of the re-
31 dehydrated sample at RT, which mechanical response agrees with the response
32 of the native dehydrated samples ('DEH'): 21.67 ± 0.67 GPa and 0.95 ± 0.03
33 GPa for E and H, respectively (Refer to table 2 for comparison). In the case of
34 frozen and defrosted several times, there is no observable extra damage in the
35 mechanical properties after the first cycle, as it can also be seen in figure 3a
36 and 3b.
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40 '[Insert figure 3a and figure 3b about here]'

41 42 43 **5. DISCUSSION**

44
45 It is well known that the fluid for storing dental tissues can alter their
46 mechanical properties [5, 10]. Alcohol, e.g. produces shrinkage of the tissue
47 because it increases the levels of interpeptide hydrogen bonding [10]. This
48 leads to enhanced hardness and mechanical stiffness, compared with the case
49 of storage in HBSS. HBSS, on the other hand, has a major percentage of water,
50 with the necessary ions to prevent dentin demineralization [5, 10] and water
51 plasticizes collagen by breaking interpeptide bonds [5], which is translated into
52 lowered hardness and stiffness of the hydrated samples. As the results show,
53 the difference is remarkable: E and H decrease to about half and one third of
54 their values, respectively, on going from dry (alcohol) to wet (HBSS) conditions.
55 Nevertheless, the fluid influences all the tooth structure and the tendencies like,
56 e.g. local gradients, are kept even though the absolutes values differ
57 considerably.
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3 The fact that the mechanical properties were kept even after two years of
4 storage in HBSS, supports the result presented by Habelitz et al. [5] that HBSS
5 is a most suitable solution for storing teeth. However, in this study it was found
6 that it is not only suitable for short periods (two weeks) as they stated, but also
7 for longer ones (years).
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11 In addition to the effects of different storing fluids discussed above, it was
12 observed in this work that the temperature of storage is also important. HBSS is
13 an aqueous buffer solution. Water is mainly located inside the tubules, so when
14 the samples are deep frozen, the increase in volume of frozen water may
15 introduce irreversible mechanical damage in the microstructure of the dentin
16 tubules as well as in the surrounding dentin. This may explain the ~25 %
17 decrease in local mechanical properties obtained among the samples stored
18 submersed under HBSS and kept at room temperature and the ones deep
19 frozen, respectively. However, this phenomenon is not observed in chemically
20 dehydrated samples because the water is replaced by alcohol, which at -15°C
21 neither freezes nor experiences any volume increase. In contrast to our
22 findings, Moscovicha et al. [5] did not observe changes in the hardness of
23 dentin after storing it at -18°C in water. However, their chosen method was
24 macroindentations with loads of 30 kg in 2.1 mm thick disks, which may have
25 hidden the microstructural changes introduced by the swelling of solidified
26 water since due to the average size of the macroindentations, it averages the
27 contributions of peritubular, intertubular dentin and tubules, and judging by the
28 load used and specimen thickness its results may be influenced by the sample
29 holder too. Nanoindentation, on the other hand, is a powerful technique to
30 investigate biomaterials with hierarchical structure, because nanoindentation is
31 capable to measure mechanical properties in the micrometer range, allowing
32 researchers to understand and discriminate the influence of micrometer size
33 structures on the mechanical behaviour.
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41 After dehydrating the former wet samples and rewetting the former dehydrated
42 samples, the measured local mechanical properties corresponded to the initially
43 dehydrated and wet samples, respectively. It is remarkable that, independently
44 of the initial storing temperatures in the dehydrated state, the E and H of the
45 rewetted conditions fitted in with the measured properties of just wet and kept
46 at room temperature ('HBSS RT Average', in figure 3a and figure 3b) samples.
47 This is explained by the fact that initially, with the former dehydration process,
48 the water is replaced by alcohol which does not freeze nor increase its volume.
49 When again the ethanol is replaced by HBSS in the rewetting step, the resulting
50 microstructure corresponds to the 'HBSS RT' condition since freezing the sample
51 did not introduce any mechanical damage. This simple model explains why all
52 the rewetted samples respond in a similar way as immersed in HBSS and kept
53 at room temperature, independently of the temperature of storage in the
54 dehydrated state.
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59 The above observations indicate reversibility of chemical dehydration as already
60 presented by Nalla et al. [5] for the fracture resistance behaviour of dentin.

6. CONCLUSIONS

Nanoindentation is a valuable technique for studying biomaterials. Not only because vacuum environment is not a requirement, but also because near in vivo conditions are achievable allowing indentations to be done under fluids.

The several conditions investigated in this work led to the following conclusions:

- The temperature of storage is an important factor for storing teeth in HBSS, if it reaches the solidification point of water. This conclusion may be extrapolated to all aqueous fluids which preserve dentin mineralization, because it is the increase in water volume after freezing which introduces irreversible damage.
- Chemical dehydration seems to be a reversible process.
- Hank's balanced salt solution (HBSS) is a suitable fluid for storing teeth.
- The mechanical properties of dehydrated dentin considerably differ from the near in vivo condition (immersed in HBSS). In this case, the plasticizing role of water and the presence of the necessary ions to prevent demineralization in HBSS are the reason for obtaining lower mechanical properties than in the dehydrated state, where alcohol is thought to increase the levels of polypeptide bonds with the expected increase in E and H that is entailed.
- Deep-freezing not only is not needed when using HBSS for storing teeth, but even detrimental.
- Healthy teeth are necessary to distinguish the mechanical response differences among different storing conditions.

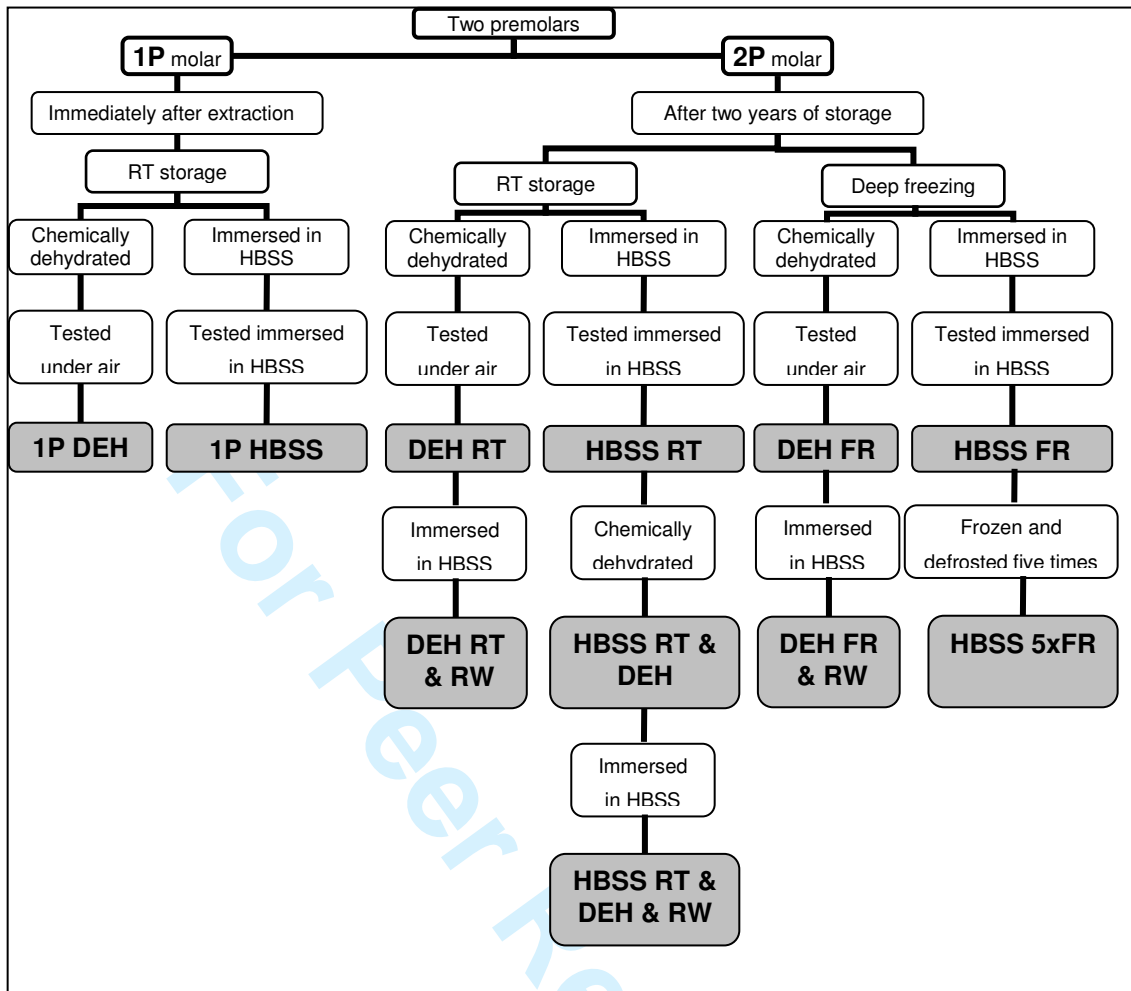
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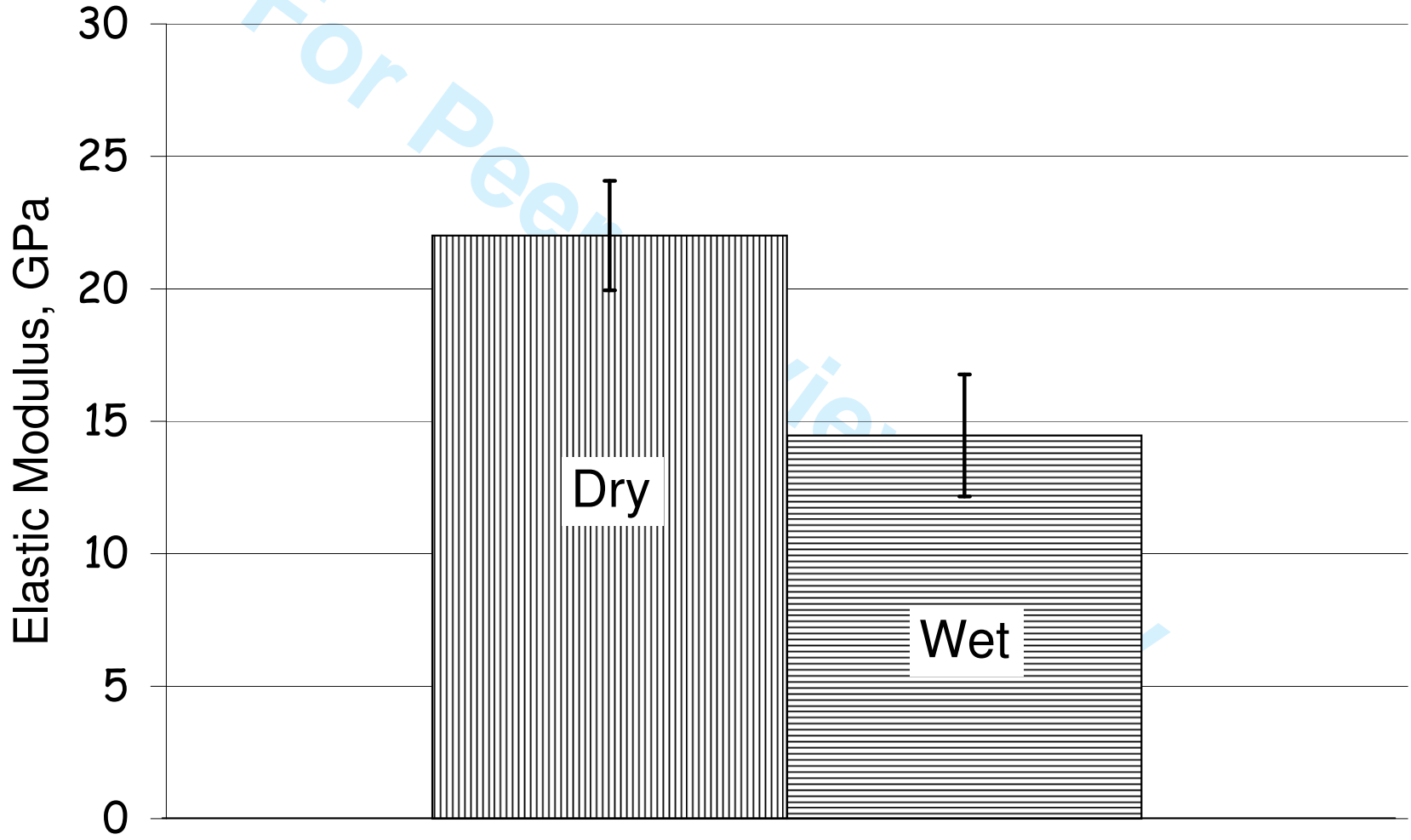
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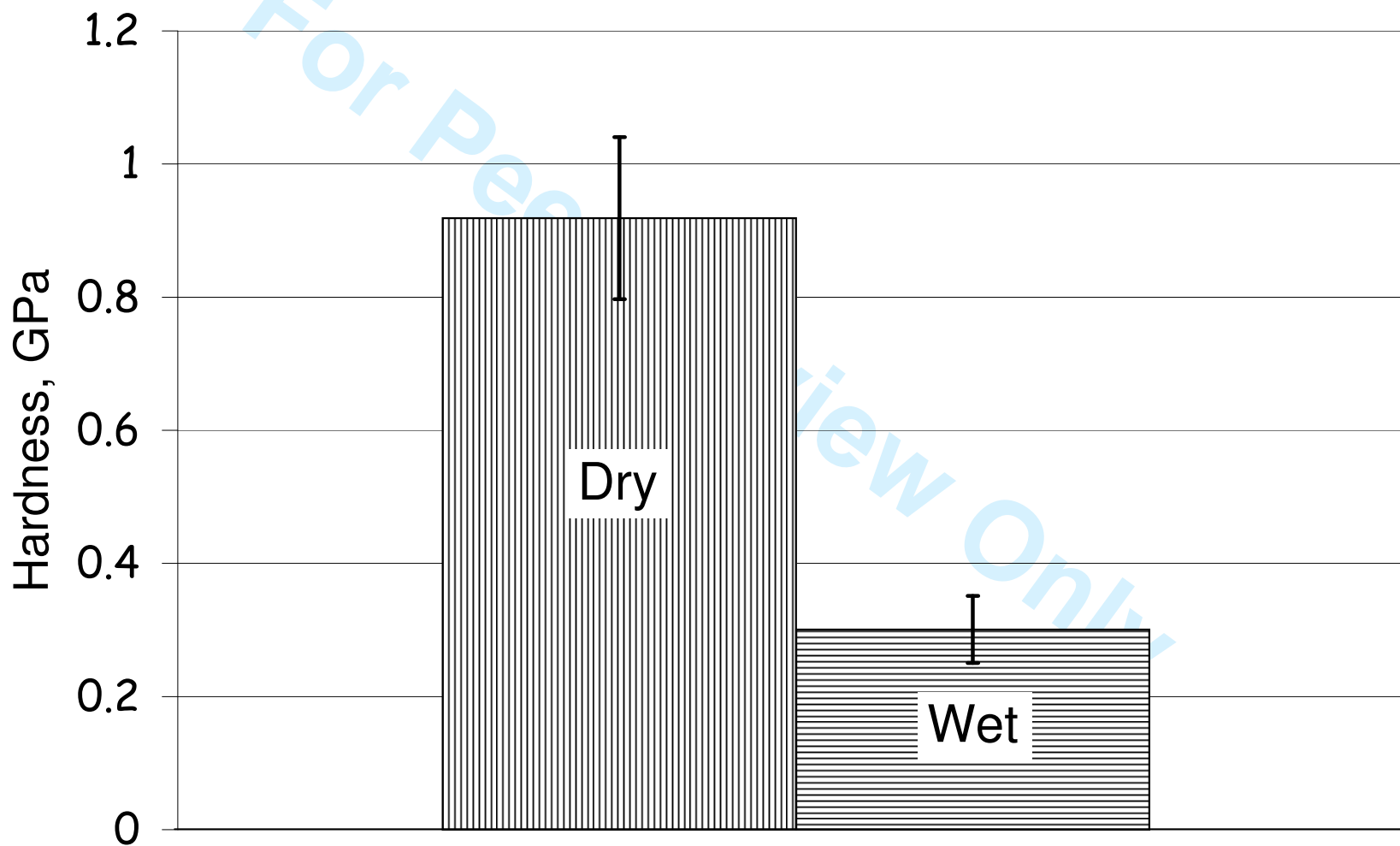


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ELASTIC MODULUS

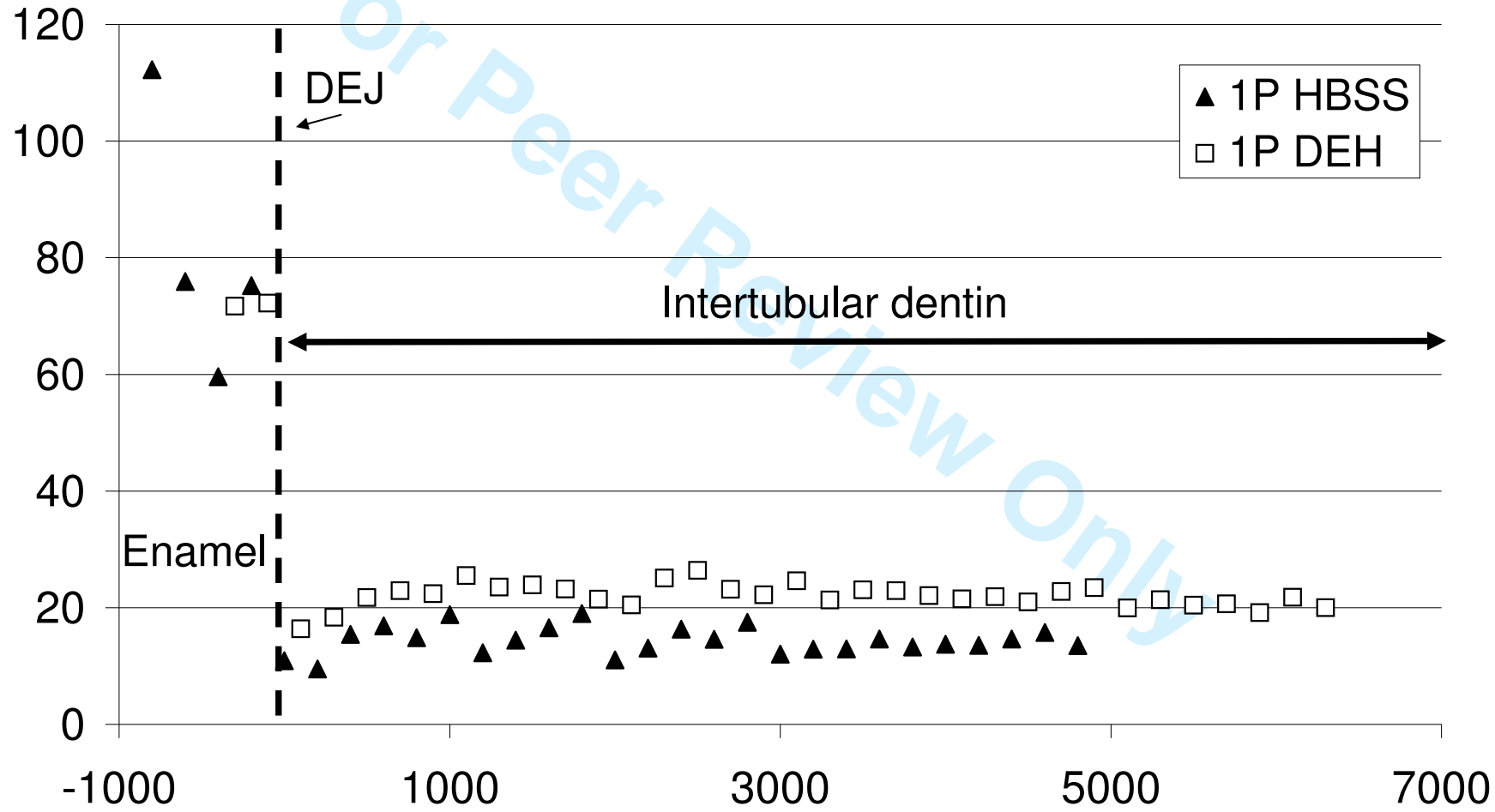


HARDNESS

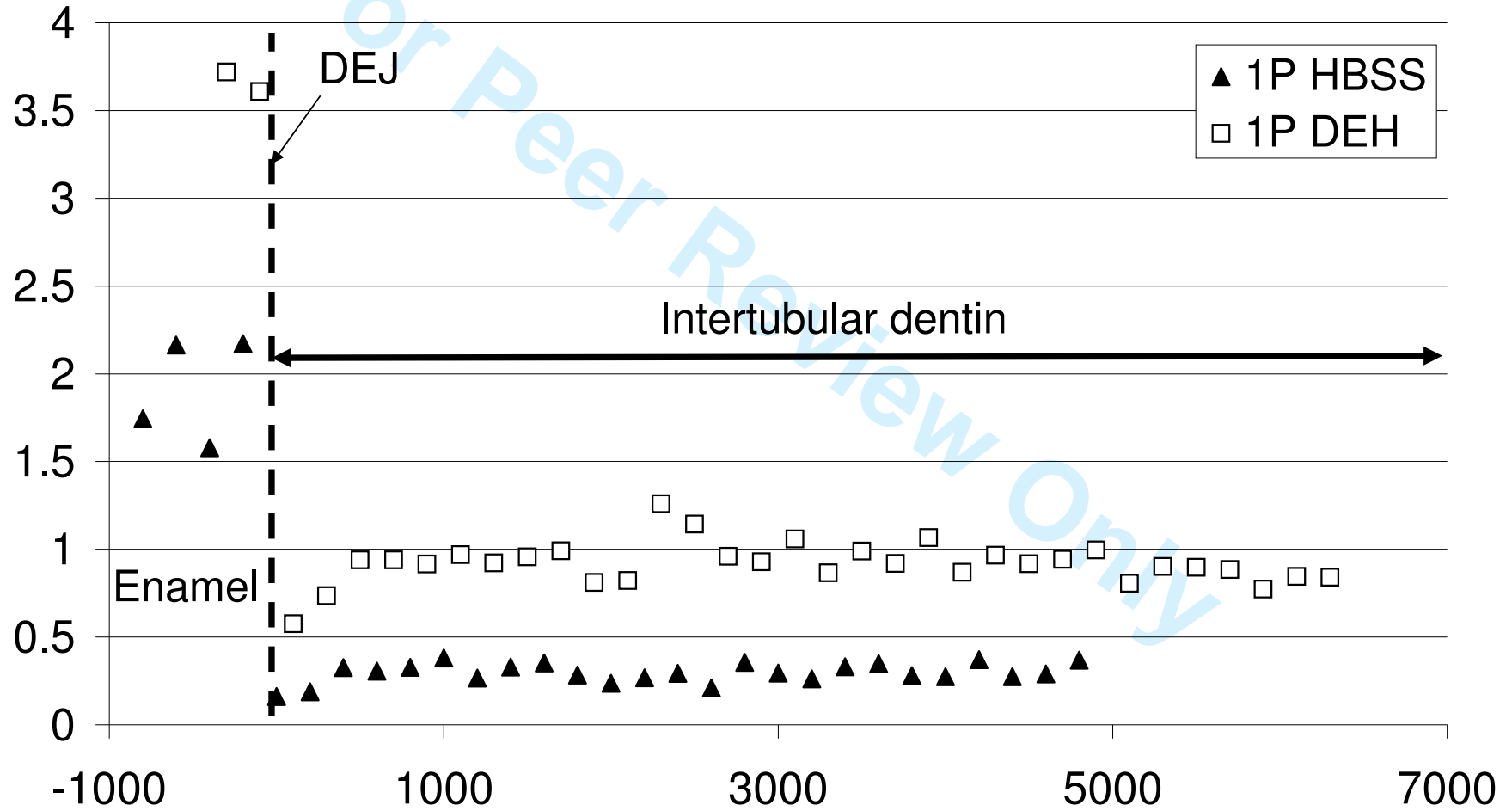


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ELASTIC MODULUS

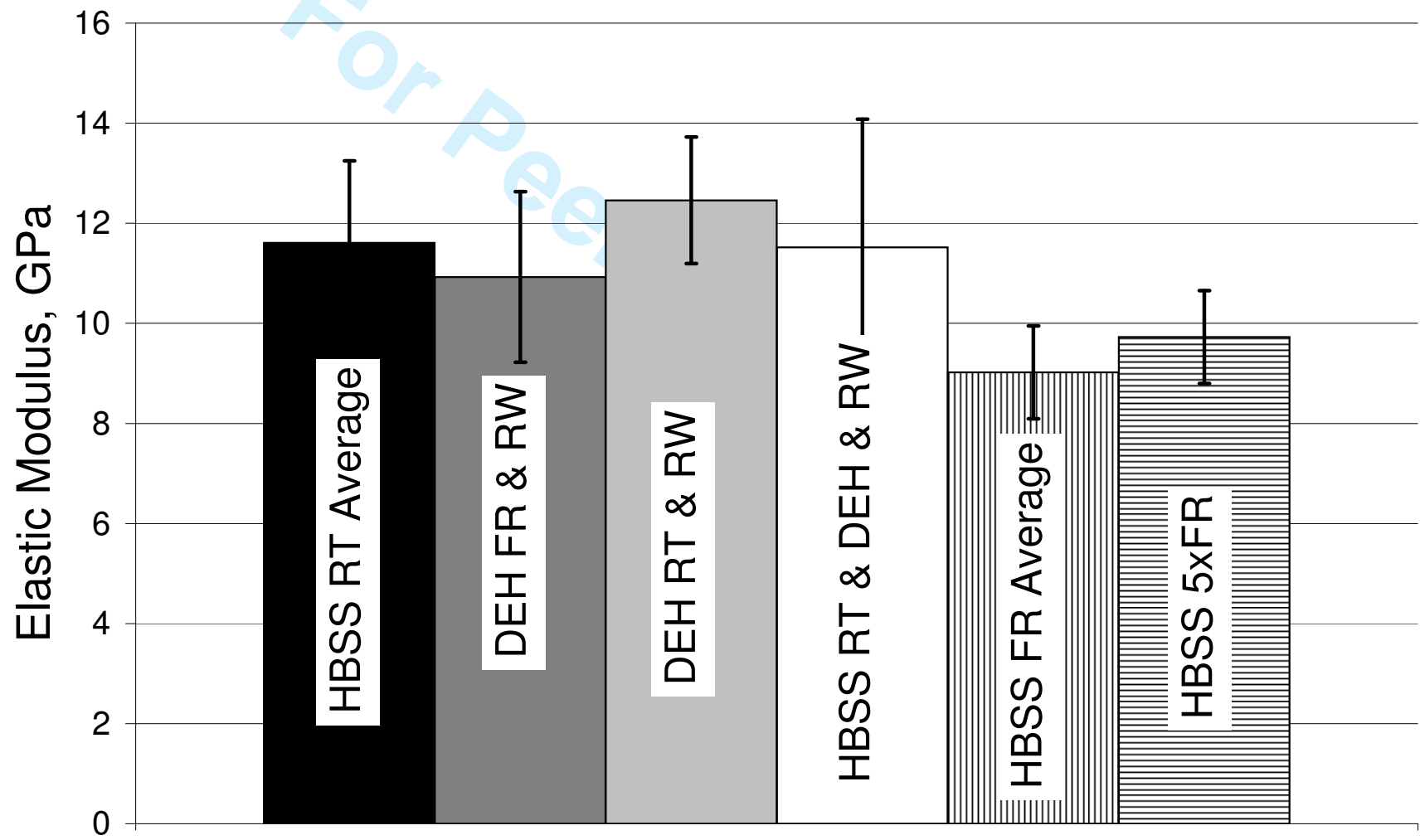


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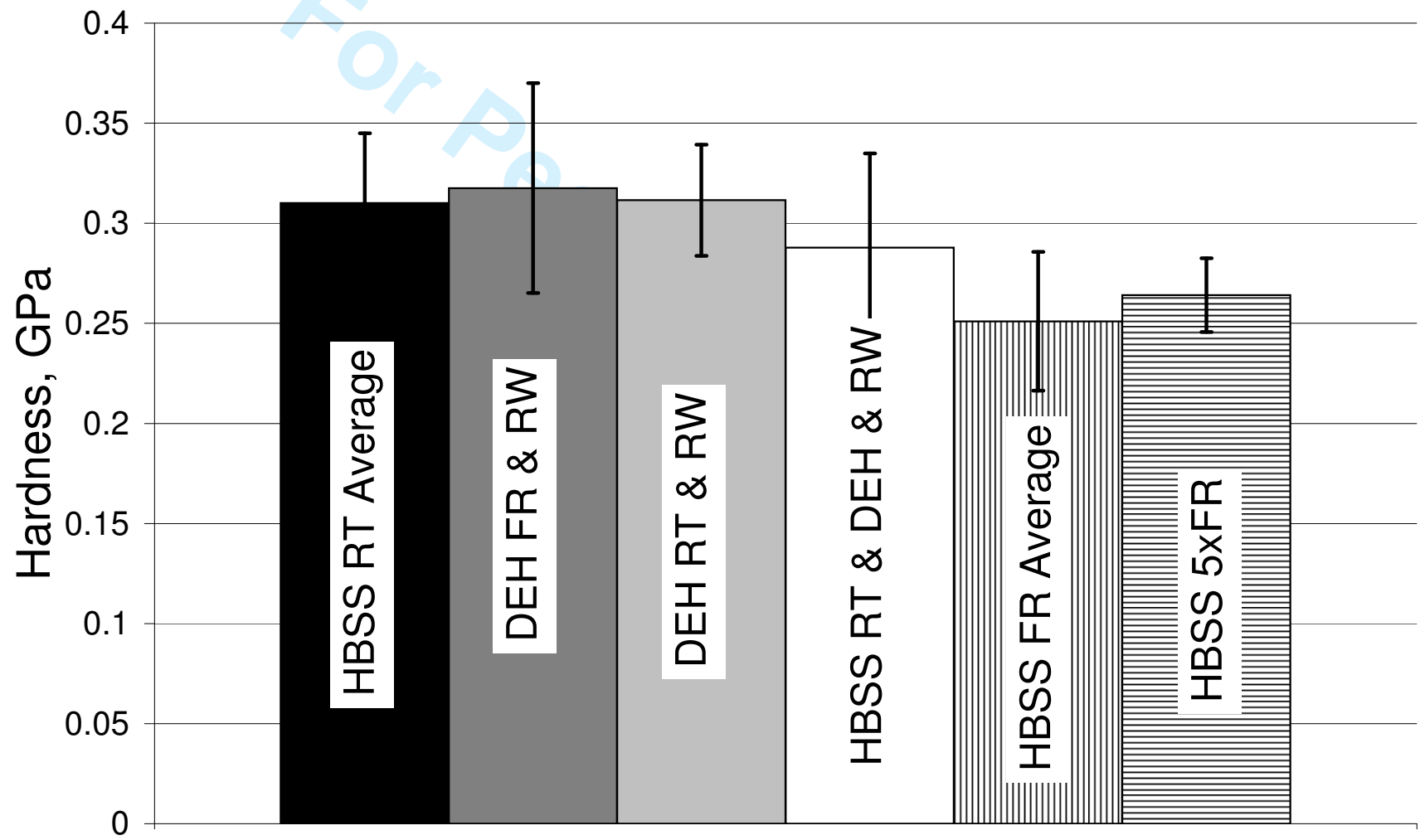


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ELASTIC MODULUS - WET



HARDNESS - WET



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TABLES

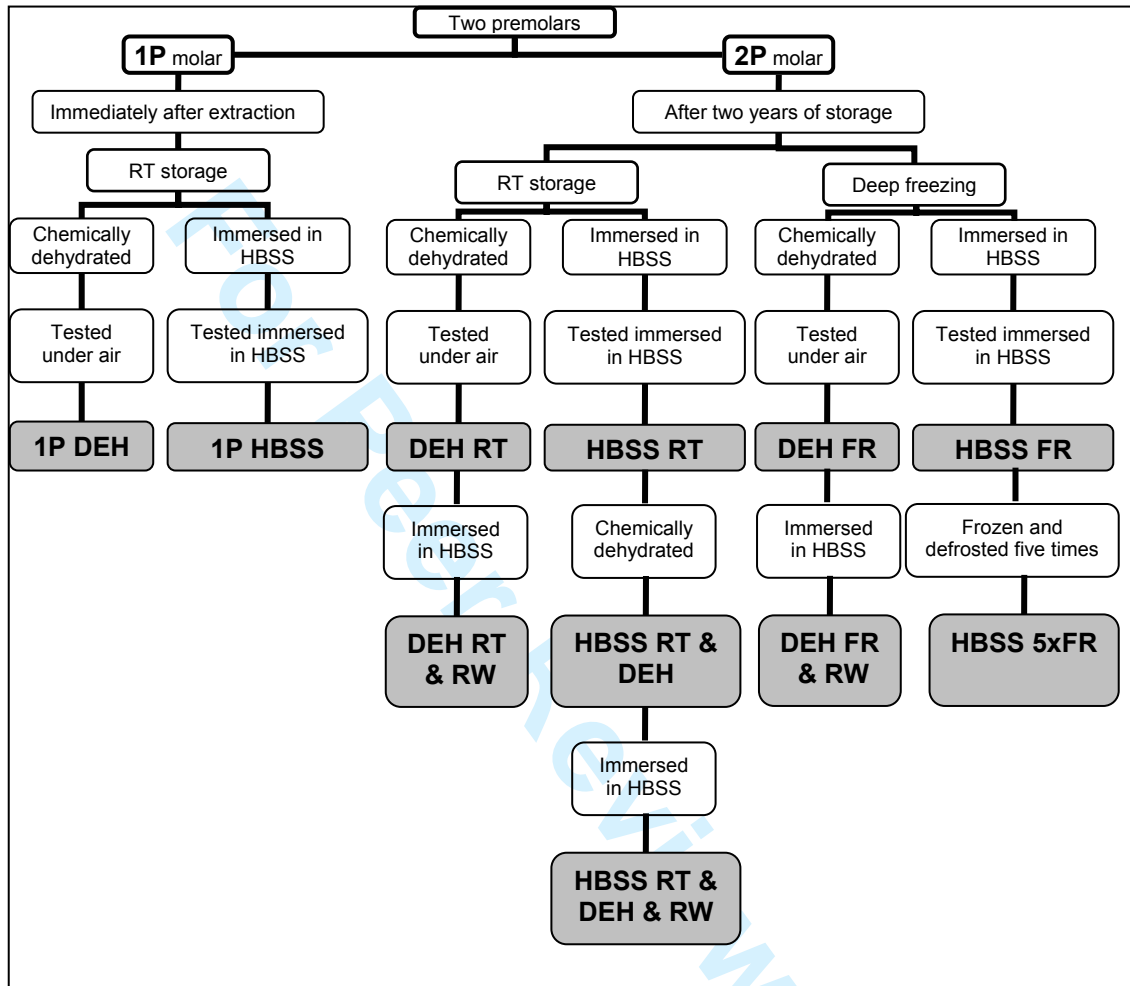


Table 1. Schema showing all the experimental conditions used (in white boxes) and the nomenclature chosen (grey boxes).

Sample	Condition		Hardness, H (GPa)	Elastic Modulus, E (GPa)
1P	HBSS		0.30 ± 0.05	14 ± 2
	DEH		0.92 ± 0.12	22 ± 2
2P	HBSS	RT	0.31 ± 0.03	12 ± 2
		FR	0.25 ± 0.03	9 ± 1
	DEH RT & FR		0.96 ± 0.07	23 ± 2

Table 2. Local mechanical properties of the initial tested conditions of both 1P and 2 P samples.

CAPTIONS

FIGURES

Fig.1. Each column represents the averaged elastic modulus (E) and hardness (H) of the 1P sample, both in the dry and wet condition. E is presented in Fig 1a and H in Fig 1b. The difference in absolute values between both conditions can clearly be seen.

Fig.2. Elastic modulus (E) and Hardness (H) of 1P sample are shown in Fig 2a and Fig 2b, respectively. In both graphs, left side of the figure corresponds to enamel, right side to intertubular dentin. Each point is a single measurement.

Fig.3. Elastic Moduli of all the tested WET conditions of 2P samples are shown in Fig 3a. On the other hand, hardness data of all the tested WET conditions of 2P samples are plotted in Fig 3b. Columns values are average values. The error bars were calculated as the standard deviation (SD) of the measurements. HBSS RT and FR average are the average during the two week of testing of each of the conditions, respectively.

FIGURES

Figure 1a

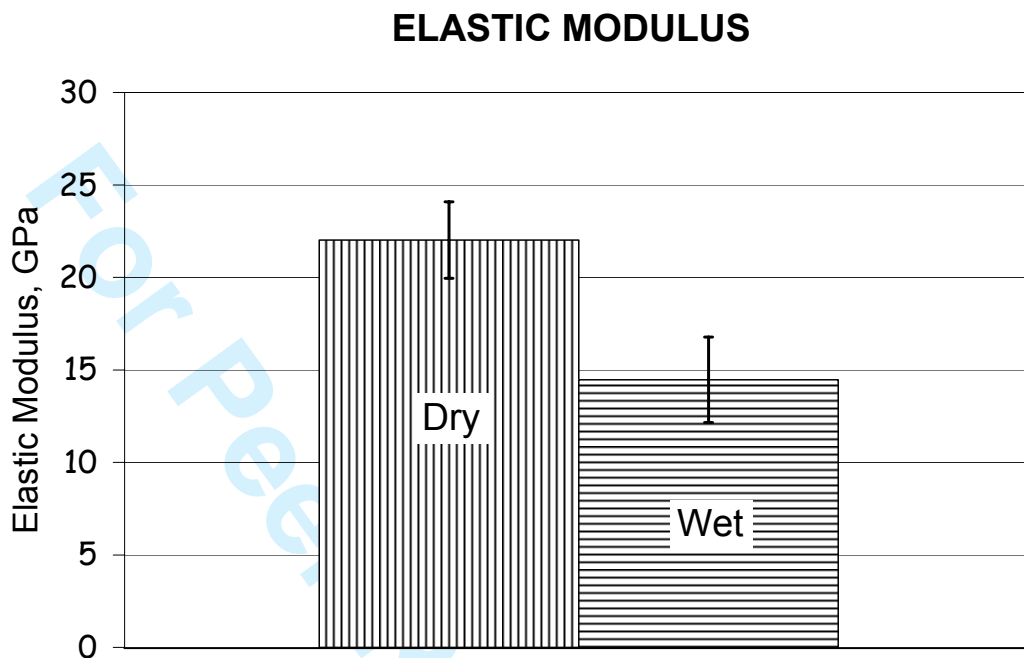


Figure 1b

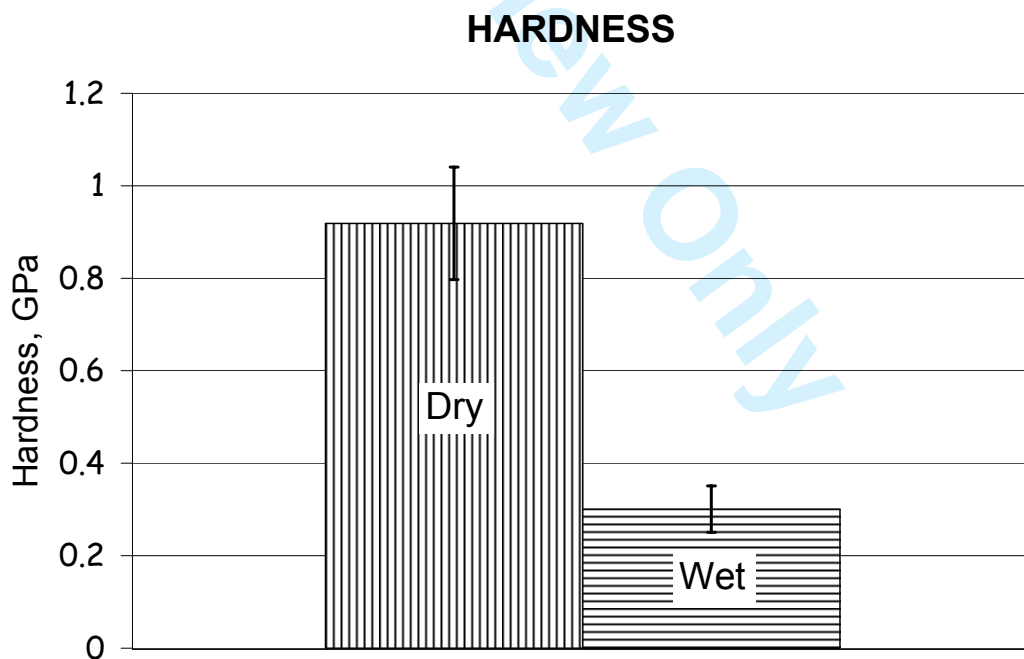


Figure 2a

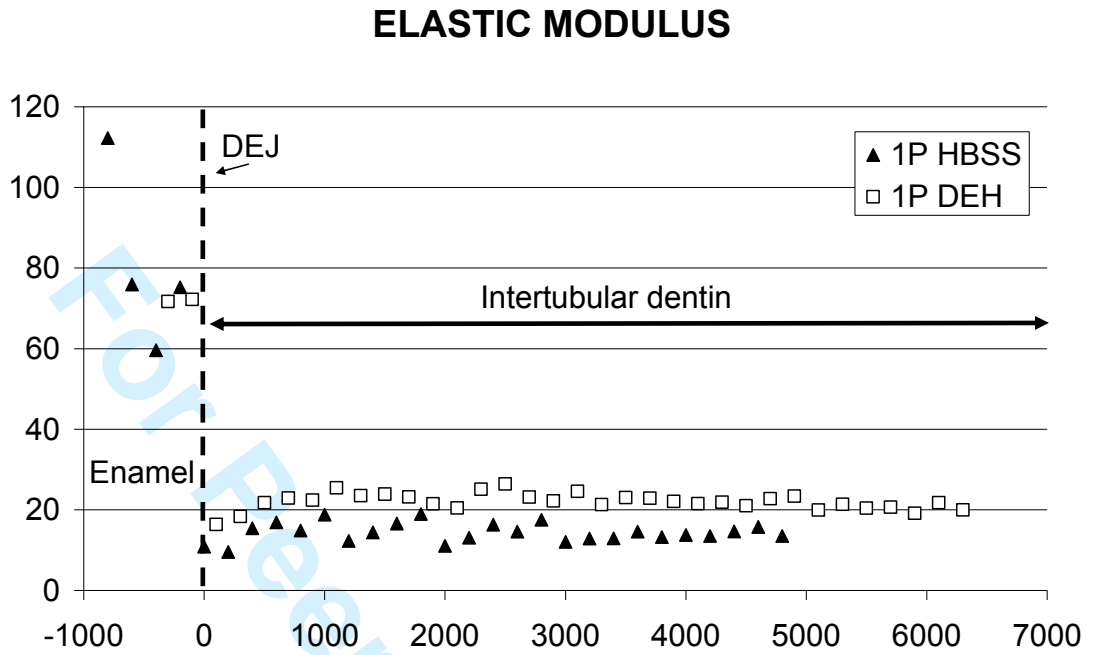


Figure 2b

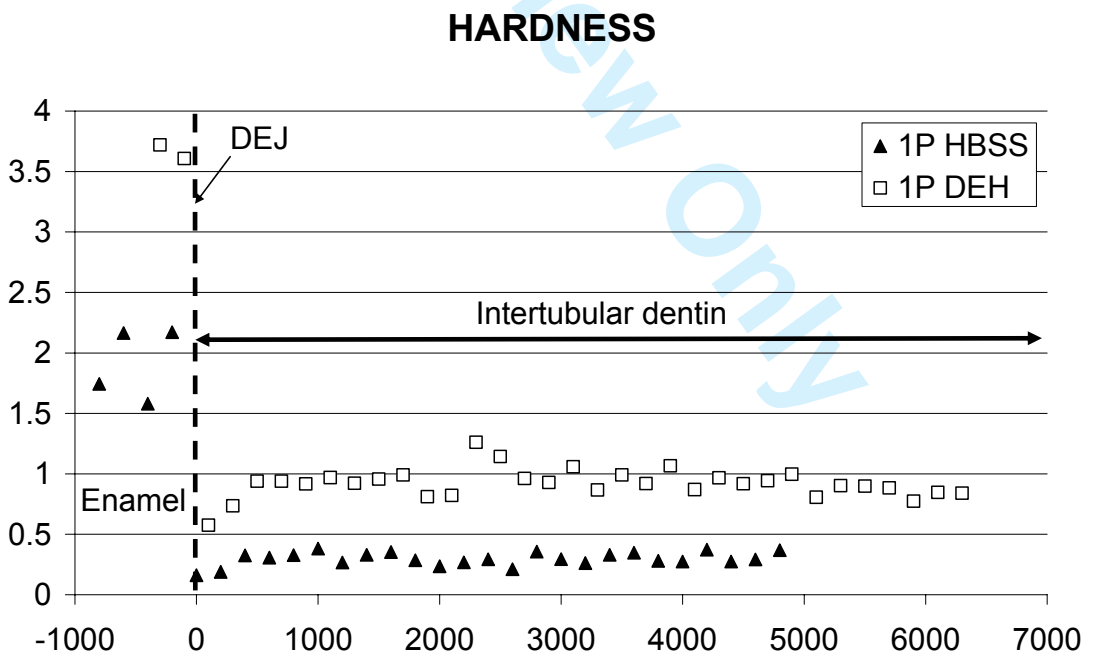


Figure 3a

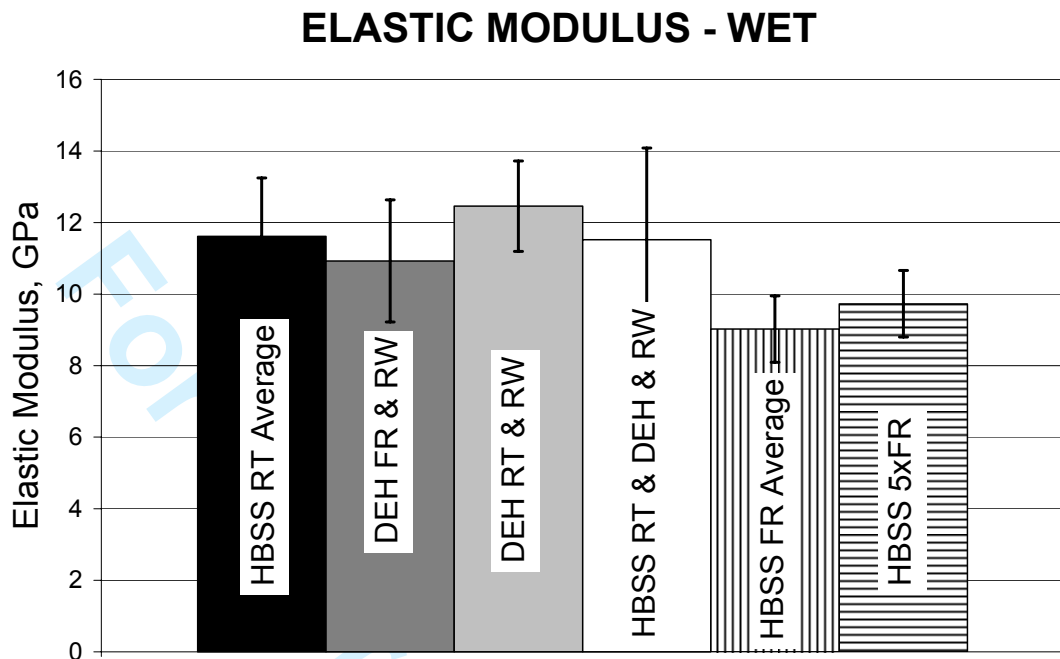


Figure 3b

