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The effects of dental grinding and sandblasting on ageing and fatigue behavior of dental zirconia (Y-TZP) ceramics

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Abstract

This study was designed to evaluate the effects of dental grinding and sandblasting on the ageing and fatigue behavior of pressureless-sintered biomedical grade Y-TZP ceramic. It was found that upon dental grinding and sandblasting, the surface of the material was heavily damaged in part plastically deformed, but the amount of transformed monoclinic zirconia was low. The partitioned tetragonal zirconia grains and pre-existing monoclinic zirconia in the ground and sandblasted surfaces hindered the propagation of the diffusion-controlled transformation during subsequent ageing. Dental grinding at a high rotation speed lowered the mean strength under static loading and the survival rate under cyclic loading. Sandblasting, in contrast, resulted in surface strengthening and substantially higher survival rate under cyclic loading. For all tested groups, about 10–15% lower survival-strength values were obtained when tested in artificial saliva, compared to dry specimens, implying that stress-assisted corrosion plays an important role in the fatigue behavior of dental zirconia.

Keywords: Dental zirconia; Ageing; Fatigue

1. Introduction

In the past decade there have been major advances in the application of yttria partially stabilized zirconia (Y-TZP) ceramics for dental restorations; this is due to their superior mechanical properties compared to other dental ceramics.[1] The replacement of traditional metal-based fixed partial dentures (FPDs) with all-ceramic prosthetic crowns and bridges has been driven by the improved aesthetics and excellent tissue compatibility achieved using tooth-colored, metal-free systems.[2] From the same reasons, zirconia full-ceramic post-and-core systems and implant superstructures were introduced to replace the traditionally used metals.[3,4]

In most cases these dental restorations are produced by the dry- or wet-shaping of ceramic green bodies, which are then sintered to a high density. In order to achieve the perfect fit between the prosthetic work and the prepared tooth structure a final adjustment by dental grinding is usually required, while sandblasting is commonly used to improve the bond between the luting agent and the prosthetic material. Because Y-TZP ceramics exhibit a stress-induced transformation,[5,6] the surface of the ground and/or sandblasted tetragonal zirconia ceramic will be transformed, i.e., constrained, as well as damaged, which will influence its long-term performance under clinical conditions. All-ceramic FPDs are manufactured to serve for a certain period of time, which normally spans 7–10 years. During functioning, these dental restorations are exposed to mechanical fatigue during mastication, which can be described as a cyclic movement of the lower jaw governed by different muscle groups in different cycle periods. The forces developed during muscle contraction act vertically on the teeth in the posterior region, and radially on the teeth in the anterior region. The mastication frequency can be as high as 1400 cycles/day,[2] while the commonly reported masticatory forces range between 342 and 1280 N.[7] In addition to mechanical fatigue, ceramic restorations are exposed to thermocycling in an aggressive saliva environment in the oral cavity, leading to stress-corrosion and consequently to a substantial drop in strength.[8,9] Furthermore, Y-TZP ceramics are prone to ageing in the presence of water. When exposed to an aqueous environment at slightly elevated temperatures over long periods of time, the surface of the Y-TZP ceramic starts transforming spontaneously into the monoclinic structure via a stress-corrosion-type mechanism.[10,11] The mechanism leading to the t–m transformation is diffusion-controlled, while the

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t–m phase transformation, once nucleated, is martensitic and is accompanied by extensive micro-cracking, which ultimately leads to strength degradation.[12] It has been reported that in 2001–2002 several hundreds of Y-TZP femoral heads failed in a short period, with the origin of the fracture clearly associated with the hydrothermal degradation; however, no such failure event for dental zirconia has been reported so far.[13] In contrast to the orthopedic community, dentists do not seem to be concerned by ageing problems, presumably anticipating that veneering and luting materials, separating the core from the oral environment and hard dental tissues, provide for a durable protection of dental zirconia against hydrothermal decomposition. However, it has been shown recently[14] that commonly used luting cements absorb water via dentine tubules, thereby exposing the zirconia core to moisture, which, in turn, may lead to ageing problems over a shorter period of time than anticipated.

This study was designed to evaluate the effects of dental grinding and sandblasting on the ageing and fatigue behavior of pressureless-sintered biomedical grade Y-TZP ceramics.

2. Experimental procedure

The material used in this study was fabricated from commercially available ready-to-press biomedical-grade TZ-3YB-E zirconia powder (Tosoh, Tokyo, Japan), containing 3 mol% of yttria in the solid solution to stabilize the tetragonal structure, 0.25 wt% of alumina addition to suppress the t → m transformation during aging, and 3 wt% of an organic binder. This material was selected for its high strength and fracture toughness and superior resistance to degradation in an aqueous environment. Uni-axial dry pressing at 147 MPa in a floating-head die was used for shaping green pellets of 20 mm in diameter and 2 mm in thickness, which were subsequently pressureless-sintered at 1520 °C for 2 h.

After firing, the disc-shaped specimens (15.5 ± 0.03 mm in diameter and 1.5 ± 0.03 mm thick) were randomly divided into groups of 10 and subjected to different surface treatments. A coarse-grit (150 μm) diamond burr mounted on a high-speed hand piece was chosen for the dry surface grinding, in order to simulate clinical conditions. The grinding load of about 100 g was exerted by finger pressure; the grinding speed was 150,000 rpm. For sandblasting, the discs were mounted in a sample holder at a distance of 30 mm from the tip of the sandblaster unit, equipped with a nozzle of 5 mm in diameter. Samples were sandblasted for 15 s with 110 μm fused alumina particles at 4 bar resulting in an (estimated) impact velocity of about 350 m/s. One group of sandblasted samples was annealed for 1 h at 920 °C, which is above the m–t transformation temperature, to re-transform the monoclinic zirconia on the surface into the tetragonal form. Before and after each surface treatment the samples were analyzed with XRD, using Cu Kα radiation. The relative amount of transformed monoclinic zirconia on the specimens' surfaces was determined according to the method of Garvie and Nicholson.[15]

Accelerated aging experiments were conducted in artificial saliva under isothermal conditions at 134 °C for 2 and 24 h. This procedure has been proposed to estimate the equivalent aging time under in vivo conditions whereby 1 h at 134 °C roughly corresponds to 2–3 years in vivo.[12] After autoclaving, the specimens were analyzed using XRD for the phase composition.

Biaxial flexural strength measurements were performed according to ISO 6872 at a loading rate of 1 mm/min, using a universal testing machine (Model 4301, Instron Corp., Canton, USA). The tests were performed both in air and in artificial saliva; in the latter case, prior to strength testing the specimens were stored for 24 h in artificial saliva at 37 °C. The surface-treated specimens were fractured with the surface-treated side under tension. The fatigue experiments were performed in air and in artificial saliva using a servo-hydraulic testing system, Fast Track 8871 (Instron, High Wycombe, UK), at a frequency of 15 Hz. A sinusoidal cyclic load ranging from 50 to 850 N was applied to the disc-shaped specimens. The lower and upper loads corresponded to a stress of 36 and 620 MPa, respectively. After 10^6 cycles the specimens were monotonically loaded to fracture.

3. Results and discussion

An SEM micrograph of a sintered, polished and thermally etched Y-TZP sample is shown in Fig. 1. The material has a dense, uniform microstructure. The quantitative microstructure analysis revealed a nearly log-normal grain size distribution with a mean grain size of 0.51 μm. The relative density of the sintered specimens exceeded 99% of the theoretical value. According to the results of the XRD analysis (Fig. 2) the material consisted of nearly 100% tetragonal zirconia. However, a significant amount of cubic phase (up to 16%) may be present,[16,17] which is hardly detected by the current standard X-ray diffraction procedure.

After dental grinding and sandblasting, however, detectable monoclinic peaks with a marked preference for the M(1 1 $\bar{1}$) orientations and a remarkable T(1 1 1) peak broadening accompanied by a reversed intensity of the tetragonal (0 0 2) (2 0 0) peaks appeared in the XRD pattern. Almost no monoclinic zirconia was observed on the surface of the sandblasted specimens that were subsequently annealed, indicating that during heat-

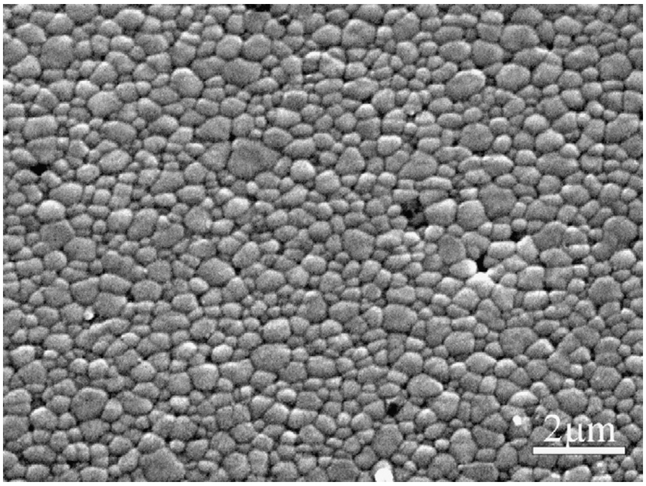

Fig. 1. SEM micrograph of sintered, polished and thermally etched dental Y-TZP ceramic.

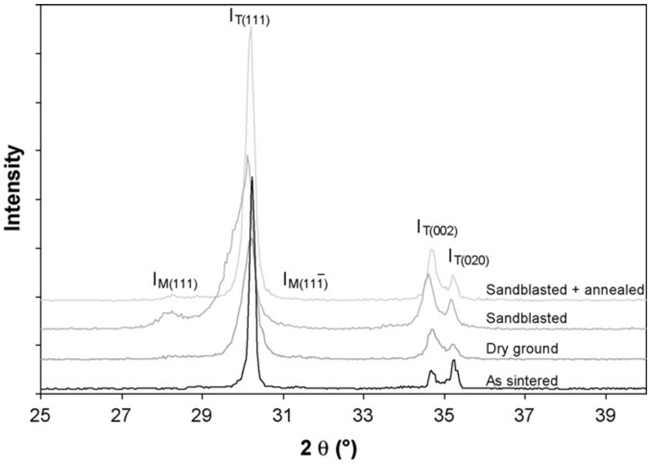

Fig. 2. XRD pattern obtained from the surface of as-sintered, ground, sandblasted and sandblasted + annealed (for 1 h at 920 °C) Y-TZP specimens.

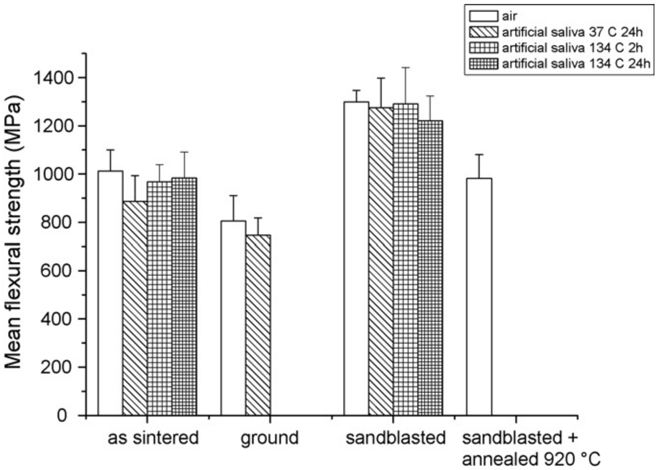

Fig. 3. Mean biaxial flexural strength values for as-sintered and surface-treated Y-TZP ceramics. (1) In air, (2) in artificial saliva, (3) after accelerated ageing in artificial saliva for 2 h at 134 °C and (4) after accelerated ageing in artificial saliva for 24 h at 134 °C. Error bars represent 1S.D. from the mean.

ing up to 920 °C the reverse m → t transformation occurred. It is worth mentioning, however, that after annealing the T(1 1 1) peak intensity and sharpness were re-established, but the reversed intensity of the tetragonal (0 0 2) (2 0 0) peaks was preserved. The relative amounts of the monoclinic zirconia on the surface of the as-sintered and mechanically treated samples before and after accelerated ageing in artificial saliva at 134 °C are listed in Table 1. Before accelerated ageing the highest amount, about 14–15%, of the monoclinic phase was found after sandblasting. A considerably lower amount, <5%, of the m phase was obtained after grinding, indicating that this surface treatment is less effective at initiating the t → m transformation in TZP materials. According to Swain and Hannink,[18] during machine grinding the tetragonal phase initially transforms to monoclinic when a grain is impacted with the diamond abrasive; this also results in severe surface disorder and copious dislocation generation. However, associated with extensive deformation and energy input, locally developed temperatures exceed the m → t transformation temperature, above which the tetragonal zirconia is thermodynamically stable and the reverse m → t transformation occurs. The latter was evidenced by the presence of numerous different t-variants within the same grain implying that the variants which were once m-phase had reverted to t-phase in a confined manner. As a result, tetragonal grains on the ground surface are partitioned and highly distorted, but the amount of transformed monoclinic zirconia is negligible. In contrast to dental grinding, at least part of the forward t → m transformation was retained upon sandblasting, indicating somewhat lower temperatures. However, according to the XRD pattern of the sandblasted samples in Fig. 2, this mechanical surface-treatment operation also resulted in highly distorted and partitioned tetragonal grains on the surface. Upon annealing, these tetragonal grains are no longer distorted, but they remain partitioned and partially aligned, with the longer *c*-axes of the t-cell normal to the surface.

With reference to Table 1, ageing at 37 °C for 24 h in artificial saliva already resulted in a detectable amount of monoclinic zirconia in the surface of the as-sintered specimens; a still higher amount of tetragonal zirconia transformed to monoclinic in these samples with accelerated ageing at 137 °C for 2 h, while with ageing for 24 h at this temperature the amount of monoclinic zirconia exceeded 25%. If we assume that the increase in the monoclinic content upon ageing was a measure of the phase instability of the Y-TZP ceramics under hydrothermal conditions, the highest instability was observed with pristine, i.e., as-sintered samples, and the lowest with sandblasted samples that were subsequently annealed above the m → t temperature. Based on these results it can be concluded that partitioned tetragonal zirconia grains and pre-existing monoclinic zirconia in the surface of the mechanically treated Y-TZP hinder the propagation of the diffusion-controlled transformation during subsequent exposure to an aqueous environment.

The results of the biaxial flexural strength measurements in air and in artificial saliva are summarized in Fig. 3. The intrinsic

Table 1
Relative amounts of the monoclinic zirconia on the surface of as-sintered and surface-treated Y-TZP ceramics before and after accelerated ageing

Material	X_m before ageing (%)	X_m after ageing (%)		
		37 °C/24 h	134 °C/2 h	134 °C/24 h
As-sintered	<1	1.5 ± 0.2	5.1 ± 0.4	25.6 ± 4.1
Sandblasted	14.5 ± 2.1	14.0 ± 1.6	14.5 ± 1.4	23.7 ± 2.4
Sandblasted + annealed 920	1.5 ± 0.5	2.5 ± 0.5	2.9 ± 1.2	9.4 ± 2.6
Ground	4.3 ± 0.8	6.3 ± 1.5	7.2 ± 2.6	24.8 ± 2.1

strength of the dry specimens of about 1000 MPa corresponds well to commonly reported values for pressureless-sintered 3Y-TZP ceramics exhibiting a comparable grain size.[16,19,20] About 15% lower strength values were obtained with as-sintered ceramics when immersed (for 24 h at 37 °C) and subsequently tested in artificial saliva, compared to dry specimens. According to Chevalier et al.,[21] this is due to enhanced stress-assisted corrosion at the crack tip by water molecules. It is interesting to note, however, that a less significant strength reduction was observed when the as-sintered specimens were subjected to accelerated ageing prior to testing in artificial saliva. Again, with reference to the results in Table 1, up to 25% of the transformed monoclinic zirconia on the surface of the specimens that were subjected to accelerated ageing in artificial saliva was not (yet) harmful to the strength under monotonic loading; besides, at least in the short-term the resultant residual surface compressive stresses seem to suppress the water-assisted stress-corrosion process. However, more systematic work is needed to confirm this assumption.

Dental grinding evidently lowered the mean strength, whereas sandblasting provided a powerful tool for strengthening, in agreement with previous results.[22,23] This counteracting effect of dental grinding and sandblasting has been explained by considering two competing factors that influence the strength of surface-treated Y-TZP ceramics: residual surface-compressive stresses, which contribute to strengthening, and mechanically induced surface flaws, which cause strength degradation. The dry dental grinding used in this work is considered to be an extremely severe process resulting in high stresses and temperatures. As shown in Fig. 4a, sharp grinding grooves were introduced into the material's surface during this process; these grooves act as stress concentrators. On the other hand, due to the high temperatures developed during dry dental grinding, the amount of transformed zirconia on the ground surfaces and hence the contribution of grinding-induced strengthening was almost negligible. Consequently, the strength of the ground materials was mainly determined by the critical defect size to initiate failure. In contrast to grinding, sandblasting is capable of transforming a larger amount of zirconia in the surface of Y-TZP ceramics, thereby contributing to the surface strengthening. Although as a result of sandblasting the material surface was heavily damaged and in part plastically deformed (Fig. 4b), the impact-induced surface flaws were not strength determining; otherwise the strength of the sandblasted material would have been reduced instead of being increased. In contrast to the present results, Zhang et al.[24] observed a slight strength degradation upon sandblasting. The reported inherent strength of the Y-TZP ceramic used by these authors was substantially higher, about 2400 MPa, than that used in the present work, indicating that the material was not only post-HIPed but also extremely fine-grained. Since there is a strong grain-size dependence of the transformability, strength and fracture toughness of Y-TZP ceramics,[6] it is likely that microstructural characteristics will play an important role in the impact-induced surface strengthening of this class of ceramics. In order to estimate the contribution of surface-compressive stresses to the overall strength, one group of sandblasted samples was annealed above the m–t transformation temperature and tested for strength. As mentioned above, after annealing at 920 °C the monoclinic content dropped below 2% and the mean strength was reduced from 1298 to 982 MPa. The difference in mean strength between the as-sintered (control) and annealed groups is statistically insignificant, confirming that sandblasting introduces surface flaws that are not detrimental to the strength of the Y-TZP ceramics, even if the residual surface-compressive stresses are released.

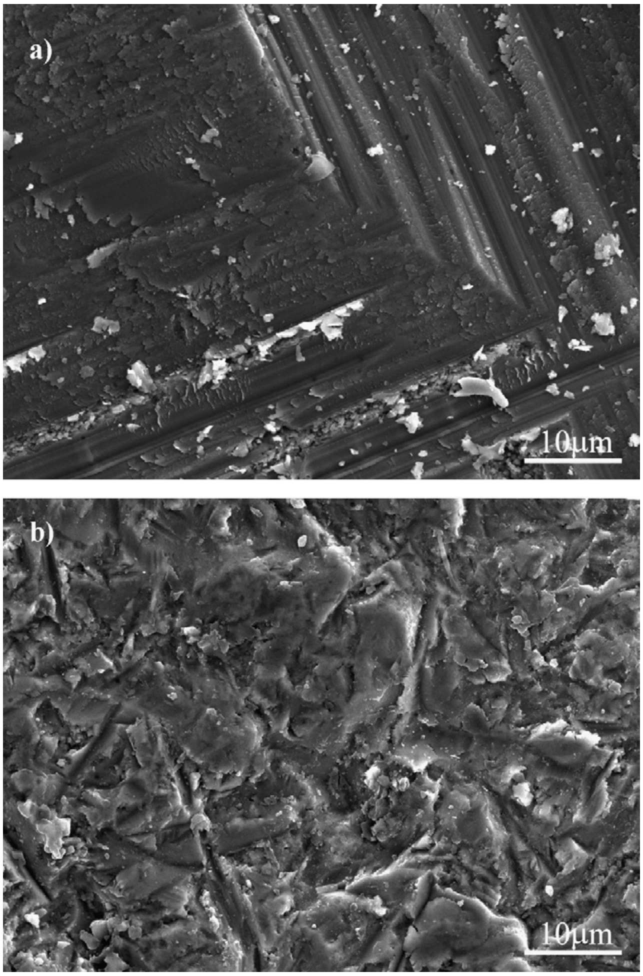

Fig. 4. SEM image showing Y-TZP surface morphology after (a) dry grinding using a 150 μm diamond burr and (b) after sandblasting.

When tested in artificial saliva, either before or after accelerated ageing for up to 24 h at 134 °C, the strength of the ground and sandblasted specimens was insignificantly lower than that of the dry specimens subjected to the same surface treatment, giving additional support to the assumption that the presence of small amounts of pre-existing monoclinic zirconia on the specimens' surface does not lead to substantial strength degradation, nor does it accelerate the water-assisted stress-corrosion process under monotonic loading.

The counteracting effects of the dental grinding and the sandblasting on the performance of Y-TZP ceramics were further verified by mechanical fatigue testing in air and in artificial saliva. The upper load limit was set at 850 N, which roughly corresponded to 60% of the mean fracture load for the control group tested under monotonic loading. According to the results presented in Table 2, the survival rate of the controls in air was

Table 2
Fatigue behavior of as-sintered and surface-treated Y-TZP ceramics in air and in artificial saliva

Surface treatment	Air		Artificial saliva (37°; 24 h)		Ageing at 134 °C; 2 h + artificial saliva (37 °; 24 h)		Ageing at 134 °C; 24 h + artificial saliva (37 °; 24 h)	
	Survival rate[a] (%)	Survival-strength + S.D. (MPa)	Survival rate[a] (%)	Survival-strength + S.D. (MPa)	Survival rate[a] (%)	Survival-strength + S.D. (MPa)	Survival rate[a] (%)	Survival-strength + S.D. (MPa)
As-sintered (control group)	64 (7/11)	1070 (69)	50 (5/10)	890 (98)	60 (6/10)	1022 (81)	50 (5/10)	877 (188)
Dry ground (150 μm)	20 (2/10)	926	10 (1/10)	940	10 (1/10)	817	n.m.	n.m.
Sand-blasted	100 (11/11)	1250 (106)	100 (10/10)	997 (330)	80 (8/10)	1287 (193)	80 (8/10)	1159 (177)
Sand-blasted + annealed	90 (10/11)	1035 (51)	60 (6/10)	888 (179)	90 (9/10)	922 (195)	60(6/10)	1004(91)

[a] After 10^6 cycles (50–850 N) at a frequency of 15 Hz.

64% and 50% in the artificial saliva solution. After grinding, the survival rate dropped down to only 20% in air and to 10% in the artificial saliva solution, whereas none of the sandblasted samples failed during the fatigue testing, neither in air nor in artificial saliva. The survival rate of the sandblasted and annealed samples was 90% in air, higher than that of the controls, but this dropped down to 60% in artificial saliva, which is the same level as that of the control group. The strength of the surviving specimens after fatigue testing in air corresponded well to the mean flexural strength of the particular group before fatigue testing. However, the survival-strength after mechanical fatigue testing in artificial saliva was found to be about 10–15% lower, with a much higher standard deviation, which is in line with the results of Chevalier et al.[21] and Morena et al.,[9] who reported on a significantly shorter lifetime for Y-TZP specimens under cyclic loading in water than in air. Notice that even lower survival-strength values and a larger variability in strength were obtained with specimens that were subjected to prolonged accelerated ageing prior to mechanical fatigue testing in artificial saliva. This holds for all groups of samples, including those that exhibited hindered water-assisted stress-corrosion under monotonic loading. The presence of pre-existing monoclinic zirconia on the specimens' surface evidently has no potential for preventing this process under prolonged cyclic loading in an aqueous environment.

4. Conclusions

The results of the present study revealed that after the application of dental grinding and sandblasting, the surface of the material is heavily damaged and in part plastically deformed. In addition, the tetragonal grains on the surface are partitioned and distorted, indicating that the reverse m–t transformation has occurred.

The highest instability under hydrothermal conditions was observed with pristine, i.e., as-sintered samples, whereas partitioned tetragonal zirconia grains and pre-existing monoclinic zirconia in the surface of the sandblasted Y-TZP hinder the propagation of the diffusion-controlled transformation during subsequent exposure to an aqueous environment.

Dental grinding at a high rotation speed lowers the mean strength under static loading and the survival rate under cyclic loading. Sandblasting, in contrast, may provide a powerful tool for surface strengthening as well as resulting in a substantially higher survival rate under cyclic loading. For all the tested groups the survival-strength after mechanical fatigue testing in artificial saliva was found to be about 10–15% lower, with a much higher standard deviation. Even lower survival-strength values and a larger variability in strength were obtained with specimens that were subjected to prolonged accelerated ageing prior to mechanical fatigue testing in artificial saliva. These results imply that stress-assisted corrosion plays an important role in the fatigue behavior, which is further influenced by ageing.

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