JOURNAL OF THE MECHANICAL BEHAVIOR OF BIOMEDICAL MATERIALS 53 (2016) 151–160

Available online at www.sciencedirect.com

ScienceDirect

www.elsevier.com/locate/jmbbm

Research Paper

A novel zirconia fibre-reinforced resin composite for dental use

Ting Wang, James Kit-Hon Tsoi*, Jukka Pekka Matinlinna

Dental Materials Science, Faculty of Dentistry, The University of Hong Kong 4/F, Prince Philip Dental Hospital, 34 Hospital Road, Hong Kong SAR, PR China

ARTICLE INFO

Article history:
Received 30 March 2015
Received in revised form
3 August 2015
Accepted 9 August 2015
Available online 17 August 2015

Keywords:
Fibre reinforced composite
Zirconia fibres
Silane coupling agent
Fracture toughness

ABSTRACT

Aims: The purpose of this study was to evaluate and compare some biomechanical properties such as fracture toughness, Vickers hardness and compressive strength of an experimental fibre-reinforced composite (FRC) filled with various percentages (0 wt%, 1 wt %, 3 wt%, and 5 wt%) of zirconia (ZrO_2) fibres.

Materials and methods: A resin matrix (78.4 wt% *bis*-GMA, 19.6 wt% MMA, 1-wt% CEMA and 1 wt % CQ) with different percentages of silanized zirconia fibres (0%, 1%, 3%, and 5% by weight of the resin matrix) was prepared. Silanization was carried out using an experimental silane blend (0.5 vol% *bis*-1,2-(triethoxysilyl)ethane+1.0 vol% 3-acryloxypropyltrimethoxysilane in ethanol, at pH 4.0). Each group of specimens was stored in two conditions – either at room temperature for one day or water storage at 37 °C for 7 days. They were randomly divided into study groups according to the test method. For fracture toughness, a notchless triangular prism (NTP) test ($n=6$) was undertaken. Hardness values ($n=6$) were measured by using a Vickers hardness testing machine and compressive strength ($n=6$) was tested. Scanning electron microscopy (SEM) images were taken at the fracture sites after fracture toughness test. The data were analysed by 1-way ANOVA (analysis of variance) and Bonferroni *post-hoc* tests ($\alpha=0.05$).

Results: The ANOVA test revealed that the experimental FRCs with 1 wt% and 3 wt% zirconia fibres showed statistically significant differences in Vickers hardness at dry condition and NTP fracture toughness after 7-day water storage, respectively. However, compressive strength of experimental groups exhibited no significant difference ($p>0.05$).

Conclusion: Silanized zirconia fibres reinforcement in resin is a novel FRC which have shown promising biomechanical properties.

*Corresponding author. Tel.: +852 2859 0303; fax: +852 2548 9464.
E-mail address: jkhtsoi@hku.hk (J.K.-H. Tsoi).

http://dx.doi.org/10.1016/j.jmbbm.2015.08.018

1. Introduction

Fibre-reinforced composites (FRC) are a relatively new material group, consisting of a plastic matrix with reinforcement by various fibres. Structurally, they are usually composed of four components: the polymer matrix, the fibres, the initiator/activator system and a silane coupling agent (with a glassy fibre material) (Vallittu, 2014).

Fibre plays a significant role to improve the mechanical properties of such a composite by transferring the stresses under an applied load from the weaker resin matrix to the stronger fine fibres. A typical initiator system for setting includes a photo-initiator, *e.g.,* camphorquinone (CQ) which is a monomer to generate free radicals that could be activated and carry out a quick polymerization (or curing) by using blue light. An essential constituent, a silane coupling agent, is used on the fibre surface if the fibres are some type of SiO_2-glass fibres. The chemical hydrogen and covalent bonds are thus built to enhance strong bonding between the resin and fibres. Consequently, silanes have become an indispensable part of FRC as they profoundly influences the adhesion strength (Puska et al., 2014; So et al., 2012).

Thermoset polymers are one of the popular polymers used in dental materials, since the molecules can join together by strong cross-links and polymer networks. It has been defined (Tsoi, 2007) that a cross-link as "a (chemical) bond, group, molecule or chain which connects two polymer chains at other than their ends." Such a cross-link could form a part of the backbone in the polymer, and thus make it more rigid and add stability. Furthermore, with two (or more) cross-linked polymers through winding and locking, eventually an interpenetrating polymer network (IPN) system would be formed (Vallittu, 2009). The polymers in the IPNs are at least partially bolstered and interweaved one by one by interpenetrating networks. However, in some IPNs, one or more linear polymers (usually with a shorter chain length) are needed to extensively penetrate into by one or more cross-linked polymers. In brief, linear polymers, which are molecules without branches or cross-linked structures, are reticulated into the cross-linked polymers and structurally could store more mechanical energy with its spring elasticity than the cross-linked polymer. Therefore, a so-called "semi-interpenetrating polymer network (SIPN)" system was formed. Surprisingly, a semi-IPN could afford more force to resist stress from outside and more widely used in FRC than the IPN system (Vallittu, 2009).

Fibres are the principal constituents in a FRC, where the fibres share and transfer the major portion of the load. Various studies (Abdulmajeed et al., 2011; Tsue et al., 2007) have provided laboratory evidence to confirm that the proper selection of the fibre type, fibre length, diameter and orientation are important to the composite. This is because most of the mechanical characteristics, *e.g.,* electrical and thermal conductivities, fatigue strength, compressive strength and modulus, of the composite are influenced (Zhang and Matinlinna, 2012). In dentistry, FRCs have become a fashionable and clinically interesting technology that could offer a new affordable option for both patients and clinicians. In fact, in addition to periodontology and prosthodontics, FRCs can be used as a root canal post in endodontics (Perea et al., 2014). In prosthetic dentistry, indirect restorations, such as a 3-unit bridge, are feasible and it is economical for patients and furthermore, usually one visit is enough. FRCs also can be applied in orthodontics for retention and as denture baseplate reinforcement (So et al., 2012). However, due to the different properties of various fibres, researchers have made many efforts in order to put diverse FRCs into practical and clinical use (Meiers and Freilich, 2006; Shinya et al., 2009; Vallittu and Sevelius, 2000). Gradually, different kinds of fibres, such as E-glass fibres, are used in FRCs today.

Zirconia, a ceramic biomaterial, is used widely nowadays in dentistry. In particular, the properties such as good biocompatibility (Lung and Matinlinna, 2012; Mallineni et al., 2013), osseointegration (Zhang and Matinlinna, 2012) and its in general high strength (Shenoy and Shenoy, 2010) have allowed zirconia to be used in various applications, such as crowns and bridges (Perea et al., 2014), implant fixture ("screw") and abutments (Tuusa et al., 2005), and orthodontic brackets (Keith et al., 1994). Nevertheless, due to its inertness and chemical stability zirconia cannot not be etched easily like porcelain (Lung and Matinlinna, 2012) using hydrofluoric acid, and some more harsh conditions such as heat (Liu et al., 2015) and much longer duration (Lee et al., 2015) might be necessary. Resin-to-zirconia or porcelain-to-zirconia adhesion represents different aspects (Liu et al., 2013). To tackle this, efforts has been put such as using laser (Liu et al., 2013), resin-infiltrated coatings (Liu et al., 2014) and zirconate primers (Cheng et al., 2014) to improve adhesion. Perhaps interestingly, not much attention has been paid to zirconia fibres. Considering the well-established chemistry (Lung et al., 2012), ease of use, biocompatibility and various well-known applications in dentistry, silanes are still dominating and might be one of the best choices for zirconia adhesion improvement. One of the promising silane developed by Matinlinna et al. (2013a), Matinlinna et al. (2013b), which is so-called a novel silane system, has shown an improved performance in resin-zirconia adhesion under laboratory tests such as water storage and thermo-cycling. On the other hand, there is little information how such silanization might affect zirconia fibres. The use of FRC in the oral environment

Table 1 – Chemicals used for the resin matrix.

Material	Manufacturer	Lot number	City and country	Purity
bis-phenol A-glycidyl methacrylate (*bis*-GMA)	Accu-Chem industries Inc.	23823	Melrose Park, IL, USA	AR
Methyl methacrylate (MMA)	Accu-Chem industries Inc.	1122	Melrose Park, IL, USA	AR
Camphorquinone (CQ)	Accu-Chem industries Inc.	A0077555	Melrose Park, IL, USA	≥ 99.0%
N,N-cyanomethyl methylaniline (CEMA)	Accu-Chem industries Inc.	T20100224	Melrose Park, IL, USA	≥ 99.5%