



Silica polymer bonding of stressed silica grains: An early growth of intergranular tensile strength

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HIGHLIGHTS

- Micro-damaged amorphous silica grains under stress release silicic acid into near contact water.
- Within 3–4 weeks above initial concentration of 300 ppm, silicic acid polymerizes and forms polymer bridges between the grains.
- The force at rupture in a single polymer branch reaches up to 0.03 mN.
- The cumulative force at rupture of the polymer network between the grains reaches 1–1.5 mN.
- The latter value is 2–3 times higher than an analogous capillary bridge force.

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ABSTRACT

Laboratory tests on microscale are reported in which millimeter-sized amorphous silica cubes were kept highly compressed in a liquid environment of de-ionized water solutions with different silica ion concentrations for up to four weeks. Such an arrangement simulates an early evolution of bonds between two sand grains stressed in situ. In-house designed Grain Indenter-Puller apparatus allowed measuring strength of such contacts after 3–4 weeks. Observations reported for the first time confirm a long-existing hypothesis that a stressed contact with microcracks generates silica polymers, forming a bonding structure between the grains on a timescale in the order of a few weeks. Such structure exhibits intergranular tensile force at failure of 1–1.5 mN when aged in solutions containing silica ion concentrations of 200- to 500-ppm. The magnitude of such intergranular force is 2–3 times greater than that of water capillary force between the same grains.

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1. Introduction

The phenomenon of aging of granular soils has long been observed both in the laboratory and in the field. It consists in a time-dependent stiffening and strengthening of granular soil in saturated conditions over engineering time-scales. Several manifestations of aging were identified over the years. Anderson and Stokoe³ found that coarse sands exhibit a low-amplitude shear modulus that

increases linearly per log cycle time. In saturated sands, normalized tip resistance after blasting have been seen to increase with time by up to 18%, whereas local friction decreased by 39% in the same time period.¹¹ Under high pressure at room temperature, clean sand was found to have developed significant intergranular adhesion bonds.³³ A time-delayed increase in stiffness of sand under sustained load was observed in dynamically compacted sands and is attributed to static fatigue.³⁶ Field observations at Jebba Dam project in Nigeria showed a substantial increase in penetration resistance in sand, weeks after blasting or vibrocompaction, even though density changes in soil have completed much earlier.³⁹ Similar results were observed in laboratory settings where cone penetration resistance

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of loose sand increased with time after blasting.¹⁶ Also in the laboratory, Hueckel et al.^{24,26} have shown a nearly 50% increase in stiffness of saturated core samples from 1000 and 3000 m depth subject for an over a 14 day aging under an *in situ* stress.

A hypothesis has been well-established that in dry granular soil, creep is a dominant aging phenomenon over engineering timescales. Based on field and laboratory results, it was postulated that a granular soil undergoes macrolocking, due to which grains become more efficiently packed under load, and microlocking, with surface roughness creep causing an increase in sliding resistance.^{35,47,31,8} Loading tests performed on an assembly of discs simulating a granular soil indicate a non-coaxiality of stress and strain-rate tensors and free rotating movements of particles under load.¹⁷ The presence of fines in dry sand is found to additionally increase creep strain and the aging rate confirmed via a discrete element model.^{50,49}

However, in submerged conditions, the increase in penetration resistance of sand is higher than in dry conditions, which has been speculated to be due to dissolution and precipitation of salt and possibly silica.³¹ Precipitation of salt upon drying has been observed to cause a substantial increase of cohesion of granular material.¹⁴ Other sediments (chalk) also exhibit a delayed compressibility changes.⁴¹

The actual mechanisms of soil aging in submerged conditions still remain a puzzling topic. Various hypotheses have been proposed with less than preponderant supporting evidence. One of an early proposed hypotheses, supported by a laboratory evidence¹⁵ was that silica precipitates and forms a thin film of silicic acid gel on top of undisturbed silica layer which acts as cementing bonds that provide adhesion force between sand grains see also.^{39,31,24,22} Experiments showed that a thin film of silanol and silicic acid chains would develop on amorphous silica surfaces in the presence of water,⁴⁸ see also.^{9,10} During prolonged loading of saturated sand, it is found that continual dissolution of minerals and some precipitation of carbonates and silica would occur.⁷ A study of silica dissolution in contact region between mica and quartz surfaces proposed that corrosion pits developed on quartz surfaces might allow a porous silica gel layer to be deposited.²⁰ There is no clear understanding of the role of the presence of biofilms on soil stiffness. Oedometer tests conducted on dense Ottawa sand showed no influence of biofilms on the ultimate shear strength of the sand,⁴² whereas Banagan et al.⁴ claims that an addition of biofilms for a few days has caused a statistically significant increase in the shear strength of Ottawa sand.²² Hu and Hueckel had recently proposed a coupled chemo-mechanical mechanism through which granular silica sand stiffens over engineering timescale under compression in the presence of water. The mechanism has been motivated by findings of Tada et al.⁵² that microcracks and micro-granulation generated near the stressed intergranular contacts an increase in the silica specific surface area, enhancing the silica dissolution by 25%. Tada et al. postulated that the dissolved silica may cause supersaturation locally and precipitate and polymerize to form

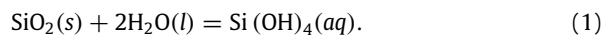
silica gel network that bonds neighboring silica grains together, thus stiffening the soil on a macroscopic scale. Indeed, preliminary experiments by the writers confirmed the presence of silica microstructures up to a few hundred micrometers in length growing near the stressed silica contacts in submerged conditions after 3 to 4 weeks of aging. Atomic Force Microscopy (AFM) pulling tests indicate that such silica polymers have tensile strength of the order of 100 nN.²¹

The objective of the current effort is to identify specific mechanism through which chemical reactions (spontaneous or induced) in geomaterials affect their macroscopic properties of strength and deformability.^{25,23,12,13} We have extended for that purpose our previous work,²¹ with the silica polymers observed growing near stressed contact regions of amorphous silica grains after 3 weeks in a liquid environment rich in silica ions. The main hypothesis behind this objective is that the outgrowth of the silica polymer provides with time the interparticle bonds that generate macro-scale cohesion responsible for the observed effects of aging. To limit the time of aging in laboratory, the fluid in the near contact environment was artificially brought to a sufficiently high concentration of silica ion (500, 400, 200 and 100 ppm), hence requiring only a limited amount of ions to create a saturated medium via dissolution to induce precipitation and gelation.⁴⁶ Specifically, isolated silica aggregates of size in the order of 10 nm would form after a week in a supersaturated solution, while after two weeks or more a significant size gel network is observed.²¹

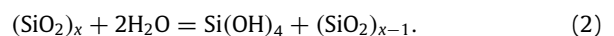
This paper presents new experimental evidence that links growth of silica polymers in engineering timescale correlates to the increase in tensile strength of the bond formed between two amorphous silica grains under prolonged compression in the presence of water. The size of silica polymers growing around stressed contact regions between two grains and their tensile strength are quantified. The overall increase in intergranular tensile strength of silicic granular material is estimated.

2. Background

Silica dissolution occurs in the presence of water via the following chemical reaction:³⁰



Because the soluble form of silica on the right hand side of the equation contains only one silicon atom, it is often named monosilicic acid. The most common form of monosilicic acid, as found in amorphous silica and crystalline silica, involves one silicon atom coordinated with four oxygen atoms. OH^- ions act as catalyst in the hydration and dehydration of silica:



An early laboratory data on silica solubility indicate the solubility of SiO_2 in crystalline silica as 6 ppm.³⁰ More recent data show that the solubility of quartz at 25 °C is around 10 ppm.⁴⁴ The solubility of anhydrous nonporous amorphous silica oxide (SiO_2) is 70 ppm at 25 °C.^{28,18} However, most common amorphous silica exists in very small particles,

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