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An innovative Setup to Investigate High-Temperature and High-Stress Flow Properties for Industrial Processes Particles

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The study of the sliding properties of coarse particles faces significant limitations in terms of the equipment developed and the theoretical frameworks employed. These limitations become even more critical when considering variables such as control of the reactant atmosphere, high temperatures and high-stress conditions. Understanding the flow property and the ability of a material to move freely without creating obstructions is crucial in various industries, including steel production. During the synthesis process, pellets containing oxidised forms of iron undergo gravity flow and progressive remelting to produce metal pellets. In the reduction phase, harsh conditions induce phenomena such as fragmentation, softening, cohesion, and changes in surface properties, all of which can hinder the natural movement of particles. Therefore, it is essential to replicate these process conditions on a laboratory scale to comprehend how these phenomena adversely affect material flow properties. To address this need, an experimental setup capable of simulating harsh process conditions and accurately quantifying parameters to characterise particle flow properties was developed.
The validity of the new setup was confirmed by comparing data obtained from it and traditional equipment using sand as a reference material. The consistency of the results validates the quality of the data obtained, highlighting the potential and reliability of the new apparatus, with the intention of extending this potential to fields of industrial interest.

1. Introduction

Conventional testers often cannot provide flow properties relevant to the application due to their inability to accurately replicate process conditions and maintain an adequate amount of material in the sensitive region of the apparatus, especially in the case of coarse particles. An example of harsh conditions can be found in steel production, a sector currently undergoing a significant transformation to reduce CO_2 emissions and promote sustainable, environmentally-friendly technologies by focusing on the direct reduction of iron ores using hydrogen gas (Boretti, 2023; Li et al., 2021). In this process, iron ore particles (iron oxide) react with hydrogen gas, producing pure metallic iron and water vapour as by-products (Choisez et al., 2024), as described by the parametric reaction in Eq (1):

$$\mathrm{Fe}_x\mathrm{O}_y + y\mathrm{H}_2\,(g) \rightarrow x\mathrm{Fe} + y\mathrm{H}_2\mathrm{O}\,(\mathrm{g}) \tag{1}$$

The reduction process chemically removes oxygen from iron oxides in a blast furnace where iron ore pellets descend while gas flows upward. A common issue faced during this process is the sticking of pellets (Di et al., 2019), which refers to the undesirable adhesion between them. During the process, the pellets are exposed to continuous reactions and extreme conditions, including high temperatures reaching up to 950 °C and an estimated compressive stress of above 800 kPa. As a consequence of these severe conditions, phenomena such as cohesion, sintering (Yi et al., 2013), and alterations in crystal structure leading to higher interparticle adhesive forces (Zhang et al., 2011) and agglomeration (Shao et al., 2011), which negatively affect the flow properties of iron ore pellets. To mitigate these issues, it is essential to study the bulk flow behaviour of the pellets under real process conditions. Due to these conditions, standard instruments have proven unsuitable for characterising this type of material. The dimensions involved are challenging, and current instruments are limited in their temperature capabilities and their compression conditions, which typically only handle a few kilopascals.

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Attempts to conduct shear experiments at high temperatures were made using conventional shear cells heated either before or during testing by using preheated samples (Smith et al., 1997) or by placing a translational cell in an oven able to reach 950°C (Kanaoka et al., 2001) or 850°C (Maarup et al., 2014). Also, rheometers and shear cells were adapted for non-ambient temperature conditions. Zimmerlin et al. (2008) developed a rheometer, based on a viscometer design, to measure the torque required for rotating an impeller, composed of a thin shaft with orthogonal pins, within a cylindrical cell heated by a coaxial furnace up to 700 °C. Ripp and Ripperger (2010) modified a Schulze annular shear cell to achieve temperatures lower and higher than ambient by introducing a heat transfer medium flow through a double casing beneath the bottom ring cell (internal volume of approximately 900 mL). Conductive plates ensured uniform temperature distribution, while the lid featured thermal insulation for cooling and electric resistances for heating. Shear tests at temperatures between -80 °C and 120 °C were conducted. Tomasetta et al. (2013) developed the High-Temperature Annular Shear Cell (HT-ASC), featuring a ring bottom cell (internal volume of approximately 85 mL) with a lid electrically heated and thermally insulated using ceramic material and a cooling water casing. This system, integrated with a Schulze Ring Shear Tester bench, enabled operation from ambient temperature up to 500 °C. More recently, Anton Paar GmbH developed a commercial shear cell with temperature and humidity control. This device consists of an annular shear cell (small cell: 4.3 mL; large cell: 18.9 mL) positioned within a heat transfer system that combines convection and radiation, allowing temperature control from -160 °C to 600 °C (Barletta et al., 2019).

As noted, standard equipment is designed to characterise fine particles, resulting in shear cells that are small in size. Furthermore, none of these instruments can replicate the process conditions, such as temperatures around 1000 °C and compressive stresses of several bars. To address these limitations, the University of Salerno (UNISA) developed a pioneering, high-temperature, gas-shielded shear cell specifically designed to accurately analyse the bulk flow of iron ore under the exact temperature and gas composition required for reduction.

2. Apparatus

A unique Reacting High Temperature Shear Tester (RHT-ST) developed at the University of Salerno (Figure 1a) can operate at high temperatures under a controlled atmosphere and is specifically designed to test iron ore pellets under various reduction conditions. The measurement principle in designing the new experimental apparatus is akin to those defined for fine particles. The system comprises a shear cell with two fundamental components: the finned lid and the trough. While the lid ensures the distribution of the force applied to compress the sample material, the fins facilitate the creation of a shear plane at a specified depth. The trough is rotated at a speed of approximately 0.015 rpm.

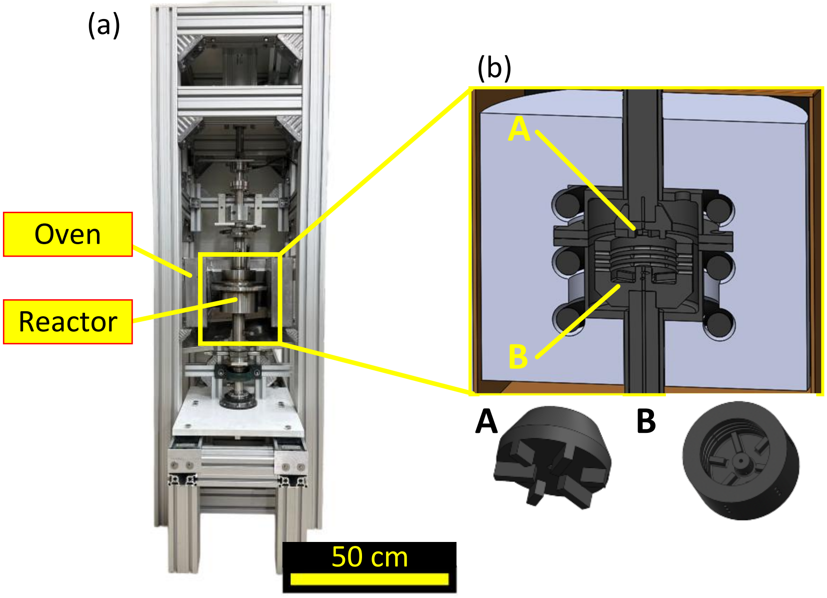

Figure 1: (a) Reacting High Temperature Shear Tester; (b) cross-section view of the reactor inside; component A refers to the lid, and component B represents the trough.

A key feature of the RHT-ST is the increased thickness of the fins arranged radially on the lid (Figure 1b-A), enabling them to withstand high shear stresses resulting from the high compressive forces under investigation. The trough (Figure 1b-B) has a volume of approximately 245 ml and includes a distributor to ensure intimate contact between the gas and the solid material being treated. The lid is attached to a pneumatic actuator through another rod, which applies a constant normal force on the lid, with a maximum normal load generating a stress of approximately 2400 kPa. A torque sensor is fitted to the lid rod to measure the torque required to hold the lid in position during trough rotation, while a position sensor monitors the lid's movement.

The lid and trough are housed within a steel reactor shell constructed of two flanged parts that can be separated to load the cell. During operation, the shell and cell are placed in an electric oven capable of reaching temperatures higher than 1000 °C. Two axial channels at the top and bottom of the reactor extend out of the oven and contain the rods supporting the trough and lid. Water-cooled seals at the ends of these channels prevent heat transfer to the torque sensor on the upper rod and the motor on the lower rod. The sectional view of the closed configuration is shown in Figure 1b.

3. Validation of the apparatus

The usability of the equipment was validated by comparing stress results obtained with the RHT-ST against those from the Schulze tester with S Shear Cell, chosen for its capability to apply higher stresses compared to other available cells. Figure 2 illustrates the comparison between the shear stress plots generated by the two apparatuses, obtained by applying a decreasing load using sand between 0.8 and 1.0 mm as a reference material. Between each compression cycle, the tested sample was released from tension by rotating the trough in the opposite direction than in the compression step.

The curves reveal that the magnitudes of the observed quantities differ, being greater in the case of the new equipment. Furthermore, the failure condition, defined as the maximum deviation between the recorded maximum value and the steady state for a given compressive stress, attenuates as the compressive stress decreases. Consequently, the comparison was made by considering different steady-state conditions for the shear stress (τ) recorded at varying normal stresses (σ).

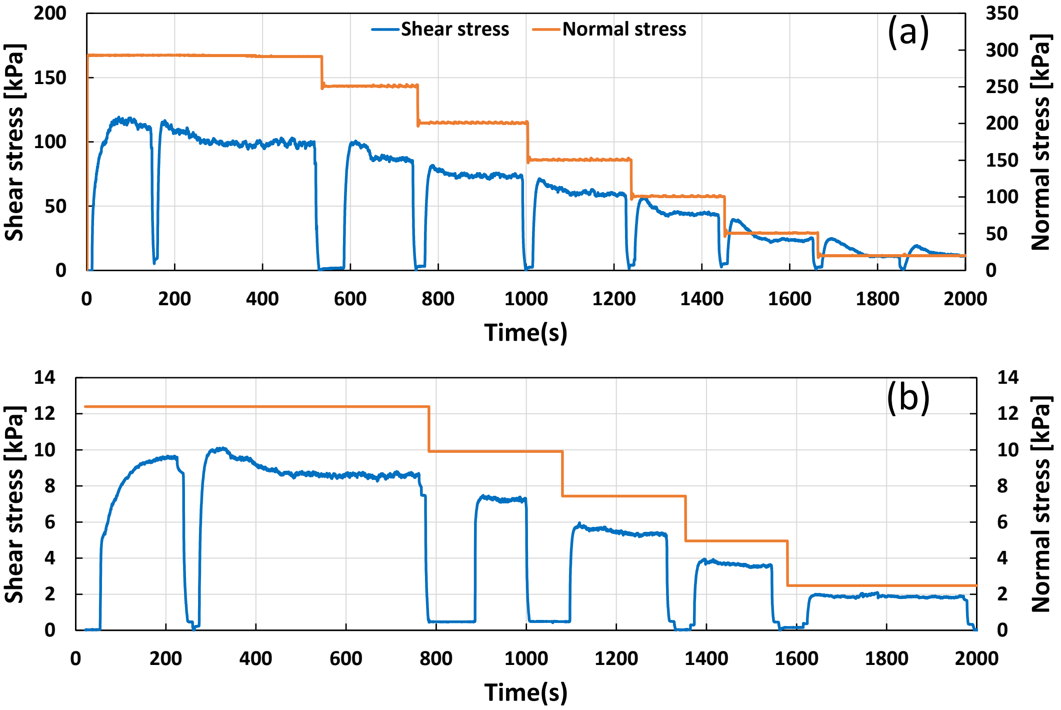

Figure 2: Experimental shear testing results obtained with (a) the RHT-ST, (b) the Schulze's tester equipped with an S Shear Cell. In orange the normal stress, in blue the shear stress.

Table 1 presents the conditions investigated and the parameters derived from the experimental data. Figure 3 presents the same data in graphical form. The curves derived from the experimental data have been approximated using a linear trend. The curve obtained from the RHT-ST is shown in orange, while the curve obtained using the Schulze tester is shown in blue.

Table 1: Applied normal compression stress and measured steady-state shear stresses in the validation experiments.

RHT-ST		**Schulze's S Shear Cell tester**	
Applied Normal Stress [kPa]	Steady State Shear Stress [kPa]	Applied Normal Stress [kPa]	Steady State Shear Stress [kPa]
305.03	118.13	12.49	7.01
263.24	102.20	10.02	5.91
213.92	85.94	7.53	4.34
163.26	66.70	5.04	2.92
113.06	46.73	2.47	1.52
63.11	26.09	0	0.17
32.04	11.14		

The high quality of the recorded signals and the consistency between the data obtained with the RHT-ST and the Schulze tester, in terms of the slope and intercept of the linear regression lines, confirm the reliability of the newly developed device for shear testing despite the different intervals analysed. The selection of sand, a material with dimensions close to those of coarse particles, ensures that the material does not deform during the compression phase, that weak interaction forces are negligible, and that there is a linear relationship between applied normal stresses and recorded shear stresses.

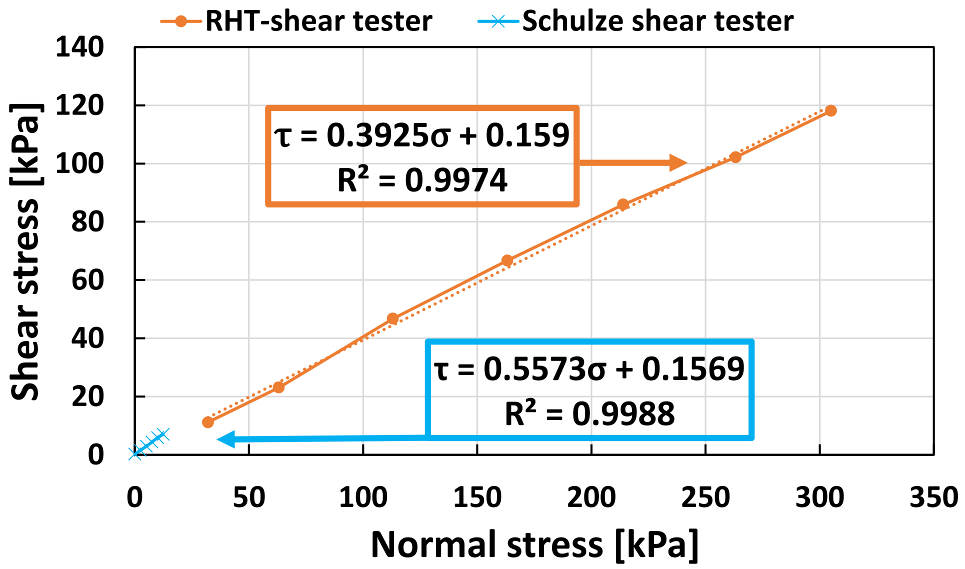

Figure 3: Graphical representation of shear testing data. In orange is the curve for the data obtained from the RHT-ST, and in blue those obtained from the Schulze tester.

4. Results and Discussion

Following the validation of the equipment, the RHT-ST was employed to characterise the same material, sand, under similar compression conditions across tests but at varying temperatures. Specifically, temperatures of 25 °C and 500 °C were examined. The reference procedure for characterisation follows the classic method defined for small particles, involving the alternation of compressions corresponding to the maximum load to be analysed, known as pre-consolidation, where a steady-state condition is reached. Afterwards, the applied load is reduced, and the point at which material creep occurs, the so-called failure condition identified by the maximum torque recorded during the load reduction phase (shear phase), is determined. This process of alternating between applying the maximum load and progressively reducing the compressive stress is repeated until the desired number of experimental points is achieved.

The stress conditions applied are presented in Table 2, along with the corresponding shear stress values calculated from the torque measured during the shear phase. The experimental plots are displayed in Figure 4. The shear stress value recorded at the powder failure during the shear phase indicates the material flow resistance. The results demonstrate that the sand resistance progressively increases as the consolidation condition decreases. This effect is more pronounced at lower normal loads. At the lowest applied normal load, there is a notable 42% increase in the material flow resistance.

Table 2: Test condition and extrapolated parameters.

Pre-consolidation normal stress [kPa]	**Applied normal stress in the shear phase [kPa]**	**Shear stress in the shear phase [kPa]**	
		25 °C	500 °C
301.76	201.68	9.58	9.76
301.78	100.91	6.56	8.41
301.81	40.81	5.34	7.58

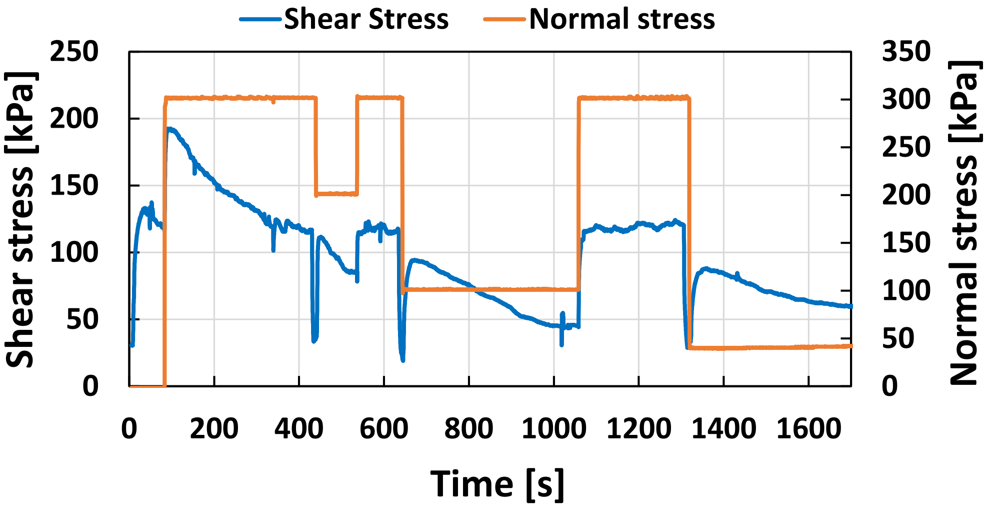

Figure 4: Tracing of the main parameters observed experimentally at 500 °C for sand powder. In orange the applied normal stress, in blue the measured shear stress.

5. Conclusions

Traditional equipment for shear testing cannot replicate high-temperature conditions and apply sufficient compressive stresses, which are critical in industrial processes such as steel production. Additionally, standard instruments are typically tailored for fine particles, making them unsuitable for materials that require larger shear cells and a more robust design. The newly developed Reacting High Temperature Shear Tester (RHT-ST) overcomes these shortcomings by enabling higher stress application and allowing accurate characterisation under extreme conditions. It also extends the testing temperature range up to 1000°C, thus effectively replicating process conditions relevant to steelmaking. The robust design, featuring thicker fins and a larger trough, makes it particularly suited for coarse particles, ensuring material integrity during testing. Preliminary tests with the new apparatus show that the results are consistent with those obtained with a Schulze Shear tester.

The RHT-ST has been successfully validated, demonstrating its capability to accurately characterise the flow properties of coarse particles, thereby addressing numerous limitations of conventional instruments. The increase in the shear stress at failure of the sand with temperature underscores the importance of carrying out shear testing of particulates under process conditions to obtain relevant flow properties. Up to now, investigations have been conducted at temperatures up to 500°C. Future applications of the RHT-ST will be extended to encompass the full range of useful temperatures and compressive conditions.

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