

POST-GENETIC EVOLUTION OF OPHIOLITE-HOSTED CR-PGE ORES: LITHOSPHERIC AND ECONOMIC SIGNIFICANCE

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Chromitite layers, pods and lenses within ophiolite ultramafic rocks are major industrial chromium sources and contain sub-economic Platinum Group Elements (PGE) enrichments. PGE and chromium are considered Critical Raw Materials (CRM) by the European Union, due to their employment in a wide range of sectors. The criticality of CRM is related to their importance for the economy, and to the risk associated with their supply.

The study of altered chromitites can give us important insights on their evolution, as well as on the behavior of precious and critical metals in contact with altering fluids. In the present work, the three main post-magmatic processes affecting podiform chromitites have been addressed: high-T subsolidus re-equilibration of olivine and spinel, low-T circulation of fluids at high (chloritization) fS_2 and fO_2 and low-T circulation of fluids at low (serpentinization) fS_2 and fO_2 . Post-magmatic processes can also affect the economic assessment of chromite ore deposits. In this study we focused on the effects of post magmatic processes on the quality parameters of ophiolite chromite sands for the foundry industry.

OLIVINE-SPINEL RE-EQUILIBRATION

Olivine-spinel subsolidus exchange is a post-magmatic process affecting chromitites down to at least 650 °C. It involves a Mg-Fe²⁺ exchange between olivine and spinel that is temperature dependent. For this reason, olivine spinel couples have been used as geothermometers and geospeedometers. Olivine and spinel compositions at grain boundaries reflect the critical temperature below which subsolidus exchange cannot proceed. Within the cores of mineral grains, in particular in the cores of larger-sized grains, minerals appear to retain their primary composition, which can be used to estimate their primary crystallization temperature. The composition of mineral rims is usually determined through arithmetic mean of several coupled analyses, within 10-20 μm from the grain boundary. As calculated $XMg = [Mg/(Mg+Fe^{2+})]$ diffusion patterns follow exponential trends (Ozawa, 1984) and estimates of the true rim compositions are very sensitive to the distance from the grain boundary.

In the present work, we developed a new approach using an exponential function to model XMg variations with the distance from the grain boundary (Bussolese *et al.*, 2019). The parameter XMg was chosen because it best represents the Mg-Fe exchange between spinel and olivine. The use of an exponential function minimizes potential errors in the calculation of re-equilibrated and primary mineral compositions, producing more reliable temperature assessments and providing insights on the geological evolution of ultramafic complexes. The approach, developed starting from the case study of Finero subcontinental mantle, has been employed with good results also for two partially serpentinized ophiolite chromitites, Iballe and Nea Roda.

Finero (Italy)

Finero subcontinental mantle is located in the Ivrea Verbano zone. Analyzed samples include fresh chromitites, chromitite-hosting dunites and chromitite-free dunites of the phlogopite-peridotite zone, from Rio Creves and Alpe Polunia localities. Diffusivity curves were modelled with an exponential function for spinel and olivine datasets, allowing the calculation of re-equilibrated (intercept on the y-axis) and primary (asymptotic value) XMg values (Fig. 1).

Temperature estimates were applied to re-equilibrated and primary XMg values following Ballhaus *et al.* (1991) calibration. The olivine-spinel re-equilibrated temperatures range between 694 and 715 °C within chromitites, between 657 and 675 °C in chromitite-hosting dunites and only a single re-equilibration temperature of 656 °C could be estimated from the chromitite-free dunite. Re-equilibration temperatures record the closing temperatures

of the subsolidus mineral exchange system. Primary temperatures represent the threshold temperatures below which diffusivity cannot maintain compositional homogeneity within the crystals (Fabriès, 1979).

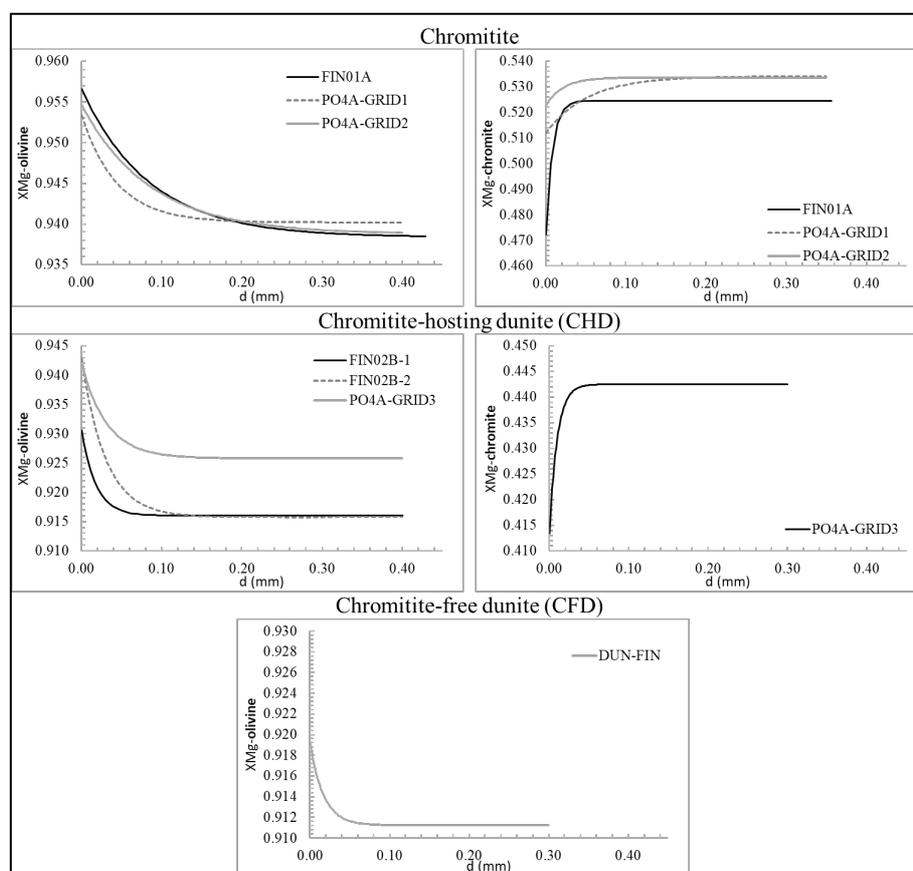


Fig. 1 - Diffusivity curves of XMg as a function of the distance from the grain boundary (d) for olivine and chromite in chromitites, chromitite-hosting dunites and chromitite-free dunites, calculated through software OriginLab.

Mg-Fe²⁺ zoning used to infer primary and re-equilibrated compositions and temperatures can also be used to estimate the cooling rate of ultramafic rocks (Ozawa, 1984). Finero cooling rates are between 10⁻⁴ °C/yr and 10⁻² °C/yr, with cooling rate curves indicating a rapid increase in cooling.

The thermal history of Finero has been established by various researcher datasets. Diffusivity profiles can add an important contribution to the reconstruction of the thermal history of the massif. Petrographic and chemical data suggest that at least two metasomatic events affected the Finero massif at different times (Grieco *et al.*, 2001). Chromitites formed during the first event in the early Permian by metasomatic interaction of basic melts with surrounding harzburgite (Grieco *et al.*, 2001).

Following the Permian event, peculiar P-T conditions caused a re-heating that re-equilibrated the U-Pb system, resulting in a large time span (Late Triassic to Early Jurassic) documented by zircon ages (Grieco *et al.*, 2001; Zanetti *et al.*, 2016). Finero cooling profiles can hence be better interpreted as due to complete re-setting of the olivine-spinel geothermometer due to the re-heating event. A long residence of the olivine-spinel system at temperatures just below the subsolidus completely re-homogenized olivine and spinel compositions. In this case, primary temperatures record the peak of the re-heating event at 849 °C, the highest primary temperature recorded by the olivine-spinel geothermometer.

Exhumation of the Finero Complex initiated during the Early Jurassic (Zanetti *et al.*, 2016). The exhumation event was rapid, bringing the complex to a shallow to mid-crustal position. The extensional activity is concurrent

to the opening of the Piedmont Ocean. Cooling rates of 10^{-4} - 10^{-2} °C/yr, recorded at Finero agree with a rapid rate of exhumation to a shallow to mid-crustal position.

Iballe (Albania)

Iballe is a small mine located in Northern Albania. Chromitite bodies are hosted within fresh dunites, with little to absent serpentinization. Analyzed samples include fresh chromitites and associated dunites. Diffusion patterns were calculated with the use of an exponential function for olivine and spinel couples of both lithologies.

The olivine-spinel re-equilibrated temperatures are 626 and 783 °C within chromitites and range between 533 and 723 °C in dunite. Primary temperatures in chromitites are 803 and 992 °C. Temperatures recorded within dunites are lower, ranging between 658 and 804 °C.

Iballe temperatures have been used to estimate the cooling rates of chromitites and dunites, following Ozawa (1984) geospeedometer. Cooling rates are highly variable between the different lithologies. Dunite samples show cooling rates comprised between 10^{-4} and 10^{-2} °C/yr, whereas chromitites have much higher cooling rates, comprised between 1 and 10 °C/yr. These different cooling rates can be explained with a different genetic history of chromitites and dunites, as suggested also by the differences in spinel mineral chemistry.

Spinel from host dunites typically have lower XCr with respect to associated chromitites. Spinel of Iballe dunites, however, show XCr values comprised between 0.8 and 0.9, whereas chromitites show lower XCr values, comprised between 0.5 and 0.6. Such values indicate that dunites have a boninitic affinity. They were formed in a supra-subduction geodynamic setting and later cooled at relatively low cooling rates. Chromitites show a MORB signature instead, and the high cooling rates are indicative of a shallow origin. Differences in spinel mineral chemistry in the area have also been reported by Qiu *et al.* (2018) and Saccani & Tassinari (2015). The former suggests a supra-Moho origin for high-Al chromitites in a supra-subduction setting, whereas the latter propose a model that explains the coexistence of MORB and SSZ-type melts through the establishment of a subduction zone close to an active mid-ocean ridge. The cooling rates inferred for Iballe dunites and chromitites could be explained by genesis of dunites in a supra-subduction setting, and later cooling at relatively low cooling rates, while the higher temperatures inferred for the chromitites fit better with a formation in a MORB setting, with consequent cooling at fast cooling rates.

Nea Roda (Greece)

Nea Roda ophiolitic rocks are located in the tectonic zone of the Serbo-Macedonian Massif, Chalkidiki Peninsula (Northern Greece). Small chromitite bodies occur to the East of the Nea Roda village. Samples were collected from a peridotite outcrop on the coast, where rare dunite dykes are enclosed within host harzburgite. The dunites host small chromitite lenses up to 10 cm thick. Chromitites have a disseminated texture with rare chromite nodules marking some of the mineralized layers. Chromitites and dunites are mostly fresh, with serpentinization limited to olivine rims. Fe-chromitization is present but not widespread.

Within Nea Roda chromitites, olivine XMg does not vary with the distance from the grain boundary (Fig. 2a), whereas chromites show a normal trend (*e.g.*, XMg decreases close to the grain boundary) (Fig. 2b).

The lack of XMg variation and diffusion patterns within olivine can be explained only by lack of subsolidus re-equilibration with the surrounding chromite, probably due to a fast cooling. In this case, the observed diffusion patterns within chromite crystals would be an artifact due to incipient Fe-chromitization at the spinel rims, or to magmatic zoning. The absence of XMg variation in olivine and the uncertainty of the XMg values at chromite rims allow only the calculation of a primary temperature using arithmetic mean of core analyses.

Estimated temperatures for disseminated chromitites range from 550 °C to 656 °C. These low temperatures could be interpreted as re-equilibration temperatures. However, the lack of diffusion patterns in olivine seems more in agreement with a limited or absent Mg-Fe²⁺ exchange, so that low temperatures are instead interpreted as the result of a genesis in a relatively surficial environment, followed by rapid cooling and consequent freeze of the exchange reaction. This interpretation is in agreement with previous studies on the area, which describe Nea Roda

as a thrust block of a section through the Moho Transition Zone, which is a zone of dunite, pyroxenite and wehrlite overlying the peridotites (Michailidis *et al.*, 1995).

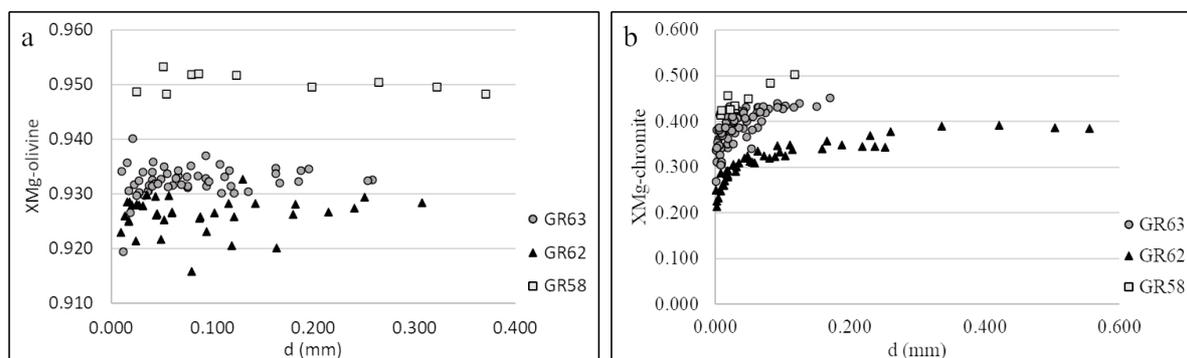


Fig. 2 - XMg variation with the distance from the grain boundary (d) in a) olivine crystals in chromitites and b) chromite crystals in chromitites.

PGE AND BME REMOBILIZATION

Low-T circulating fluids can alter primary mineralogical assemblages and induce remobilization of some elements and/or crystallization of new phases. The most common low-T alteration processes affecting chromitites are chloritization and serpentinization. The aim of the work is to study remobilization of precious metals in contact with fluids at different conditions. For this purpose, two localities showing widespread chloritization and two localities showing widespread serpentinization have been selected.

Remobilization in chloritized chromites

The two selected chloritized areas are Gomati and Nea Roda, both located into the Serbo-Macedonian Massif in Northern Greece. In the area of Gomati there are several small abandoned chromite mines, and rocks were collected from three sampling sites, St. George, Limonadika and Tripes. Nea Roda chloritized chromitites have been collected from a dump close to a fresh chromitite outcrop.

The samples are mainly massive chromitites, associated to clinopyroxenites or serpentinites. Chromite crystals are frequently altered into Fe-chromite, and all primary silicates (*e.g.*, clinopyroxene relicts) within chromitites have been replaced by Cr-chlorite.

Platinum Group Minerals (PGM) are abundant within chromitites, but none were found in the silicate host rock. They are found mainly within unaltered chromite, and rarely within altered chromite or fracture zones. Laurite is the most abundant PGM in all the sampling sites (Fig. 3a). Within Tripes, five unknown Ru-Os-Ir-Rh-Ni-Fe-Cu-rich PGM have also been detected (Fig. 3b). Tripes also shows relatively high total PGE contents, up to 3516 ppb, 9 to 20 times higher than the PGE total content of the other Gomati and Nea Roda sampling sites.

Base Metal Minerals (BMM) are abundant both in chromitites and associated clinopyroxenites. The assemblage comprises Ni-Fe sulfides, arsenides and antimonides. The only primary sulfide in Nea Roda and Gomati is pentlandite, mostly preserved within unaltered chromite grains. Secondary sulfides are godlevskite, heazlewoodite, millerite and chalcocite. No alloys were detected in the samples (Fig. 3c).

A rare suite of arsenide and antimonide minerals was found in clinopyroxenites associated to chromitites in St. George sampling site. Known mineralogical species detected were nickeline, maucherite, orcelite, breithauptite and the newly revalidated dienerite. Potential new mineralogical species detected show the following mineral formulae: Ni_5As_2 , $(\text{Ni,Cu})_{5-x}\text{Sb}_2$ (Cu-rich Sb-analogue of orcelite), $(\text{Ni,Cu})_2\text{Sb}$ and $(\text{Ni,Cu})_{11}\text{Sb}_8$ (Cu-rich Sb-analogue of maucherite).

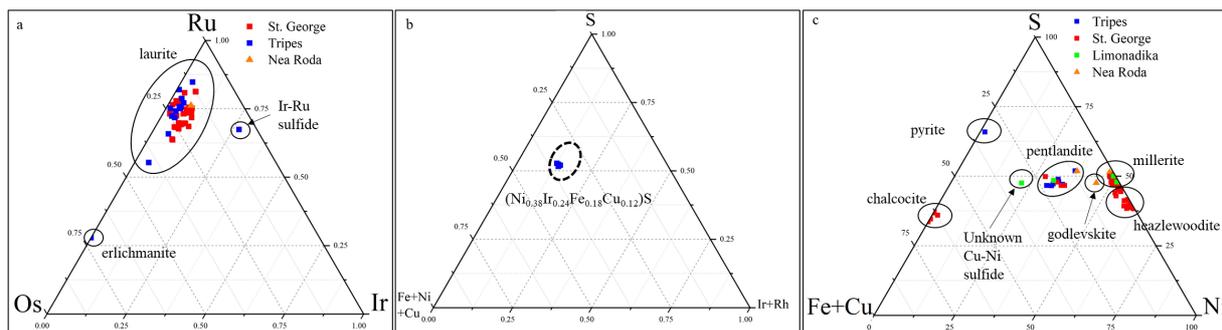


Fig. 3 - Ternary compositional diagrams (atm. %) of a) PGM in Gomati and Nea Roda, b) unknown Ni-Ir-Fe-Cu sulfide at Tripes, c) Ni-Fe-Cu sulfides in Gomati and Nea Roda.

Chromitites from St. George, Limonadika and Nea Roda show high concentration of IPGE (Ir, Os, Ru) compared with PPGE (Pt, Pd, Rh), and PGE normalized patterns typical of podiform chromitites. Tripes chromitite is decidedly different from the others, and shows Os- Ir- Ru and Rh enrichments similar to those found in the supra-Moho Nurali Level 2 and CHR-I chromitites (Grieco *et al.*, 2007; Zaccarini *et al.*, 2004). The high PGE content in Tripes reflects a higher melting degree with respect to Nea Roda, St. George and Limonadika, and could be related to a differentiation process within a magma chamber, as suggested also for other PGE-rich chromitites generated in a supra-Moho stratigraphic position (Malitch *et al.*, 2003; Grieco *et al.*, 2007).

Both Gomati and Nea Roda underwent pervasive alteration during their post magmatic evolution. The circulation of post-magmatic fluids caused the replacement of primary silicates by chlorite, the breakdown of primary sulfides into secondary ones and the crystallization of secondary arsenides and antimonides. Arsenides and antimonides in the area probably crystallized at temperatures below 600 °C due to the circulation of hydrothermal fluids rich in Sb, Pb, Cu, Au and Ag, originating from the porphyry Cu-Au of the Kassandra district, located 5 km to the North (Bussolesi *et al.*, 2020).

Secondary Ni-Fe sulfides also crystallized or replaced primary sulfides at temperatures below 556 °C (Tzamos *et al.*, 2016). The absence of alloys suggests that the circulating fluids had relatively high fS_2 , and only limited desulfurization of primary sulfides took place. High fS_2 fluids could have also been responsible for the formation of widespread chlorite and ferrian chromite.

The circulating fluids altering the silicate and Ni-Fe sulfide assemblage, however, did not affect the primary PGM assemblage. As primary PGM are mainly early magmatic phases which were included in chromite crystals, they were generally not touched by altering fluids. In the few cases where PGM came in contact with circulating fluids, however, their composition was not altered, suggesting that the fluids were at relatively high fS_2 .

Remobilization in serpentinized chromites

The two selected serpentinized areas are the ophiolite chromitites of Skyros (Greece) and Abdasht-Soghan (Iran). Skyros hosts small, non-economic chromitite occurrences associated to serpentinites, and samples were taken from the sites of Agio Iohannis and Agio Alexandria. Abdasht-Soghan consists of two mining areas. Samples were collected from Panja Metri, Omid Bekhoda, Gechin (Soghan), Site 1 and Site 2 (Abdasht). Skyros and Abdasht-Soghan chromitites are heavily serpentinized, and primary silicates are rare olivine relicts. Fe-chromitization is present in both areas, but it is not widespread.

PGM are rare in Skyros and abundant in Abdasht-Soghan, and comprise unaltered laurite, altered laurite and PGE alloys (Fig. 4a). PGE alloys are systematically associated to Fe-chromite pores, fracture zones or they form reaction zones around laurite. Whole rock PGE content is comprised between 94 and 247 ppb within Skyros, and between 16 and 420 ppb within Abdasht-Soghan. The high IPGE/PPGE ratio (4.81-19.86) is typical of podiform chromitites.

BMM are abundant both in Skyros and Gomati. The most common ones are secondary Ni-sulfides (*e.g.*, heazlewoodite, millerite), Cu alloys and Fe-Ni±Cu alloys. Primary sulfides are rare pentlandite grains and one

bornite crystal (Fig. 4b). Ni arsenides are present in both complexes but are not widespread.

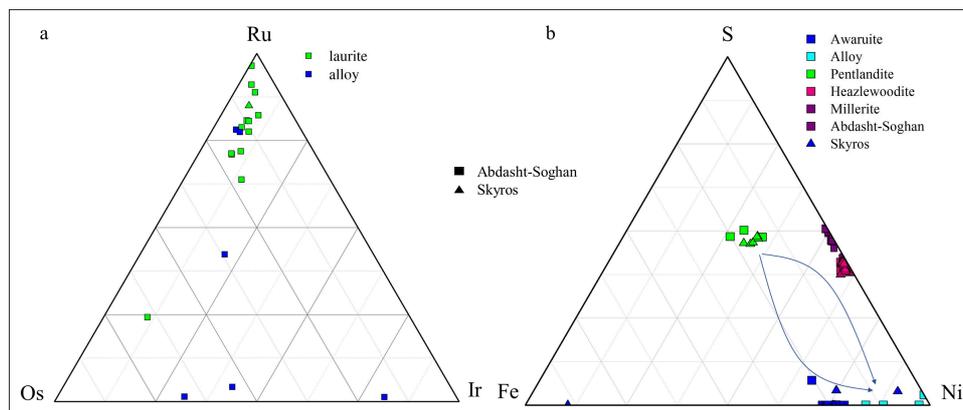


Fig. 4 - Ternary compositional diagram (atm. %) of a) BMM in Skyros and Abdasht-Soghan and b) PGM in Skyros and Abdasht-Soghan.

Primary PGM and BMM can be affected by the circulation of altering fluids. PGE and BM alloys, in particular, are the product of alteration of primary sulfides due to pervasive percolation of low fS_2 fluids during metamorphic events (Garuti & Zaccarini, 1997).

Some PGM grains found in Abdasht-Soghan have been of particular interest in the study of desulfurization processes. They show intermediate alteration stages, where primary laurite rims are S-depleted and Os-enriched. The best-preserved evidence is a polyphasic grain constituted by a laurite core, an Os-poor laurite reaction zone and an Os-rich alloy rim found in the Abdasht-Soghan complex.

This suggests a partial and selective mobility of PGE during serpentinization in the Abdasht-Soghan chromitites. Serpentinizing low- fS_2 fluids reacted with primary laurites to form PGE alloys whose composition is determined by the differential behavior of PGE. Mineralogy and texture of alloy-laurite composite grains suggests that the PGE alloy is the phase in equilibrium with serpentinizing fluids and that the reaction progresses from the rim towards the core. A mass balance calculation normalized to the Os content was performed on the partially desulfurized grain, and shows that Cu is added to the system by circulating fluids. Ir is the only PGE showing a mass increase during desulfurization, suggesting that Ir was released by other minerals (*e.g.*, primary BMM, olivine). Most of the PGE loss is due to release of Ru from primary laurite. The mobility of Ru suggests that this element has a partition coefficient between fluid and alloy much higher than both Ir and Os and can be easily released to the fluids (Grieco *et al.*, 2020). Less reliable data, due to the lower contents of PPGE, suggest that Rh is more mobile than Ru and that Pt and possibly Pd can be added to some extent to the system by fluids.

The mobility of PGE during alteration is confirmed by mantle-normalized PGE patterns, showing a double peak for Ru and Os instead of the typical single Ru peak of unaltered ophiolite chromitites. The double peak can be produced by loss of Ru during alteration events, causing a relative Os increase.

CHROMITE FOUNDRY SANDS

Chromite foundry sands, bonded with resins, are employed in the industry to form molds for casting metal and steel when a high performance of the sand is required. Chromite foundry sands have to meet strict quality parameters in order to ensure a good final product. The most important ones are Cr_2O_3 content (higher than 44%), Fineness Index (a measure of the grain size distribution of the sand, required to be between 40 and 75), SiO_2 content (below 2.5%), and Acid Demand (a buffering test devised in order to test the reactivity of the silica portion of chromite sands, required to be below 10, 8, and 6 at pH = 3, 4, and 5, respectively). Low silica content is necessary in order to avoid chemical reactions between silicate impurities within the sand and the binding resins, which could result in a decrease of the sand performance.

The European market of chromite foundry sands is dominated by South African products, and new sources are necessary in order to meet the increasing demand and to diversify the market. The present work aims to provide new insights into the production of foundry chromite grade from metallurgical-grade chromite produced from ophiolite-type deposits. For this purpose, two chromite concentrates from ophiolite deposits in Iran and Greece have been tested and compared with South African chromite sands. The first one is a sand used in the Iranian foundry market, the second one is a metallurgical-grade chromite concentrate.

The Fineness Index parameter is within the ranges of chromite foundry sands for all the tested products. Whole rock analyses on the sands revealed that SiO₂ contents are below the threshold for the Iranian sand (2.01 wt.%) and the South African one (0.70 wt.%), but too high for the Greek sand (7.72 wt.%). The Acid Demand Test (ADT) value is within the limits in the South African sand, but exceeds the upper threshold within the Greek and Iranian samples. If the high ADT for the Greek sand is to be expected due to its high silica content, Iranian AD values are more puzzling. The low difference in silica content between Iranian and South African products cannot explain the high difference in ADT values between the sands.

The only possible explanation is the different reactivity of silicate minerals in the acid environment of the test. South African sand is dominated by orthopyroxene silicates, Greek sand is dominated by an olivine-serpentine mix and Iranian sand is dominated by serpentine. ADT on pure silicate samples of chlorite, serpentine, olivine and orthopyroxene at a fixed silica content of 2.5% show a very different reactivity of the minerals (Fig. 5). For all pH values, the orthopyroxene gangue respects ADT limits, classifying as the least reactive mineral and, therefore, the best one. This explains why South African sand is the most performing one, as its gangue is mostly composed of pyroxenes. Olivine performs worse than orthopyroxene, but exceeds the ADT limits by a tiny amount. The worst performing silicate sands are chlorite and serpentine, which are highly above the ADT limits.

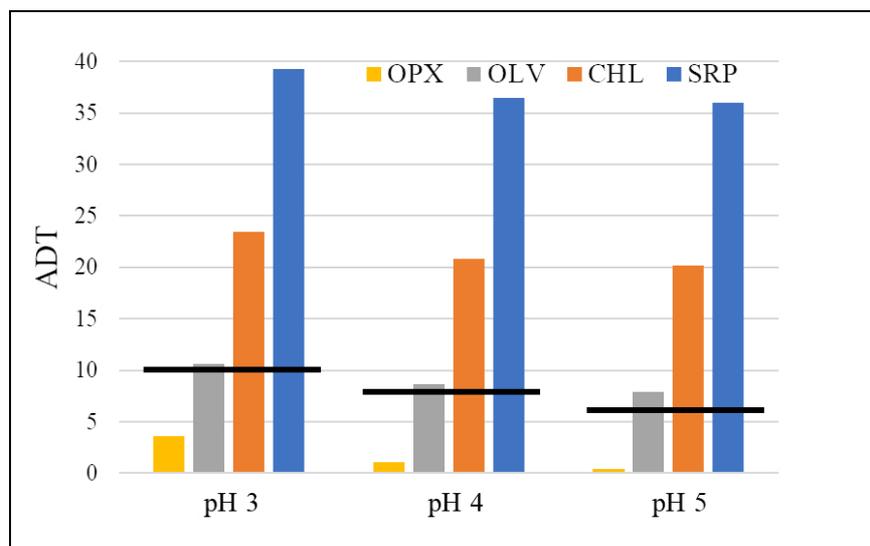


Fig. 5 - Acid Demand Test (ADT) values at pH = 3,4 and 5 of pure silicate sands (OPX: orthopyroxene, OLV: olivine; CHL: chlorite; SRP: serpentine). Black line represents the AD quality threshold.

Results show that ophiolite chromites dominated by a chlorite or serpentine silicate assemblage are not suited for the foundry market. Hence, the possibility to produce foundry sand from ophiolite chromitites is limited to those with an olivine or mixed olivine-serpentine silicate assemblage, which is anyway quite common. In order to assess the maximum serpentine content of olivine-serpentine chromite sands that can be successfully enriched till foundry sand quality, two additional Acid Demand tests were performed: *i*) olivine and serpentine mixes at different proportions but at a fixed 2.5% SiO₂ content (Fig. 6a), and *ii*) pure olivine and serpentine sands at SiO₂ contents lower than 2.5% (Fig. 6b).

Acid Demand and serpentine modal contents are not in a linear correlation, and even low serpentine amounts lead to high Acid Demand values. An ophiolite chromitite with a serpentine gangue should be purified to SiO₂ contents so low that it is not economically viable to perform such separation. On the other hand, olivine silicate assemblages can be purified to more affordable SiO₂ contents in order to meet the Acid Demand parameter.

The present work shows that the production of chromite foundry sands starting from ophiolite chromites is possible, but only for those sands where the silicate impurities assemblage is dominated by olivine, with very low or negligible amounts of serpentine.

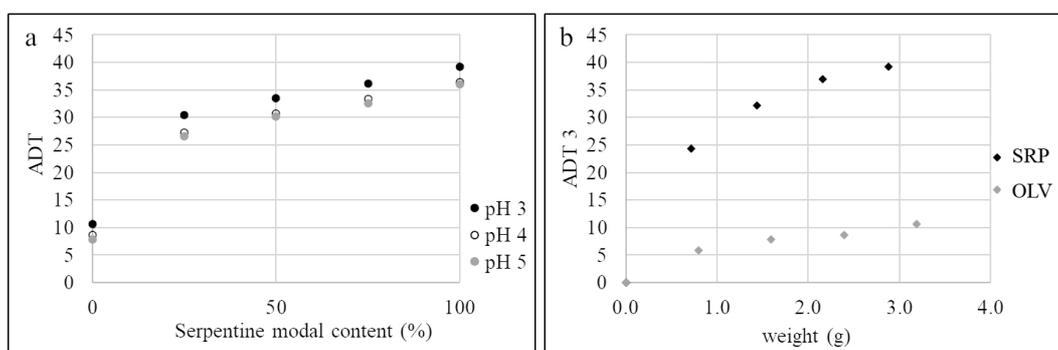


Fig. 6 - a) ADT vs. serpentine modal content in a mix of serpentine and olivine sands at SiO₂ 2.5%, at pH = 3, 4 and 5; b) ADT at pH = 3 vs. weight of material (g) for pure olivine and serpentine sands.

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