Abstract—Based on thermodynamic investigations of the reduction of lead from its sulfide compounds using the electron-donating properties of sulfur of its sulfide, we studied the effect of various factors on the reduction of lead ("metallization") in molten sodium hydroxide. In the range of temperatures of smelting PbS with the alkali (470–510°C), there occurs a "burst" metallization reaching 95% at $T = 550$°C; at $T = 650$°C, the process is completed in 15 min; at 650–500°C, the reduction of the metal occurs in the diffusion region. The NaOH : PbS weight ratio (α) exerts a direct effect on the yield of the compact metal ("lens"). At $\alpha = 1.8$–0.8, the yield of the compact metal is 96–98%. It has been confirmed experimentally that the chemism of the utilization of elementary sulfur is related to a disproportionation of the latter with the accumulation of S^{2-} and SO_4^{2-} ions and the possible formation of polysulfide sulfur S_n^{2-}.

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Based on the results of thermodynamic investigations of lead reduction from its sulfide compounds using the electron-donor properties of sulfur of its sulfide [1], we experimentally studied this process in the medium of molten sodium hydroxide.

The experiments were performed using an installation consisting of an electric furnace, steel crucible with a lid, and a block for controlling desired temperature regime. As initial materials, chemically pure (KhCh) lead sulfide and remelted caustic soda were used. The charge of PbS was 20 g. The NaOH : PbS weight ratio was varied in a range of $\alpha = 0.18$–1.8. The PbS powder was mixed with sodium hydroxide with an addition of water (7–10% of the mixture weight). The pasty product obtained was dried by slowly increasing temperature from 20 to 200°C until the complete removal of moisture (20 min). After this, the material was heated to the working temperature (350–650°C) of melting, whose duration was 2.5–40 min.

After the completion of heat treatment, the crucible was removed from the furnace and cooled to room temperature; then, its contents was leached in hot water. The solid–liquid mixture was divided on a vacuum filter; the residue was washed until the neutral reaction. The aqueous phases were integrated and analyzed for the content of NaOH, S^{2-}, and S^{6+} using known methods [2]. The deposit was carefully dried on a filter, weighed, and analyzed for the content of the sulfide sulfur by decomposing the compounds in a hypochlorite solution with a subsequent deposition using $BaSO_4$. The phase composition of the deposit was determined by X-ray diffraction using a DRON-3 diffractometer.

Figure 1 shows a sharp dependence of the degree of lead reduction ε_{Pb} depending on temperature. It is seen that an increase in T from 350 to 450°C virtually does not affect the reduction process. In the range of 470–510°C, there takes place a "burst metallization," reaching ~95% at 550°C. A further increase in temperature to 600–650°C ensures the extraction of lead into the metallic phase at a level of 100%. The character of the reduction process is confirmed by the results of X-ray diffraction analysis of the deposits obtained after leaching the fusion cakes and by the estimation of the sulfur content in them using hypochlorite reduction with a subsequent determination of their weight.

Figure 2 displays the dependence of the degree of lead reduction from its sulfide on the duration of its contact with the alkali at $T = 650$ and 500°C ($\alpha = 1.72$). It is seen from this figure that at $T = 650$°C the reduction process occurs quite dynamically and is completed after a 15-min contact between the phases. In the stage of the active phase (in the first 6 min), about 85% Pb is reduced. A decrease in the temperature to 500°C substantially affects the duration of the reduction process: after 40 min, $\varepsilon_{Pb} = 88$–90%.

Based on an analysis of curves of lead reduction related to the determination of the time of achievement of equal extractions at temperatures of 500°C (τ_1) and 650°C (τ_2) with the use of the procedure suggested in [3], we investigated the relationship between τ_2 and τ_1. From the slope of the straight line obtained, we determined that the energy of activation of the reduction process is 24.8 kJ/mol. In the temperature range of $T = 650$–500°C, the process occurs in the diffusion region.

piombo riduzione ossidi negativi con soda caustica
In questi giorni ho fatto una scoperta importante, tratta da questo documento:
http://resources.metapress.com/pdf-preview...7m&size=largest

In pratica, la materia grigia attaccata alle griglie negative, e' PbS, alias solfato di piombo, in questo documento, viene descritto il metodo per trasformarlo in metallo, con guadagni anche del 100%.

La procedura e' la seguente, e potete anche provarlo da voi, io ho provato ed e' riuscito, non posso postare foto per ora poiche' la fotografica si e' guastata.

Mettere in un crogiolo di ferro (possibilmente spesso piu di una lattina, il problema non e' la soda, il ferro non si rovina con la soda, e' il calore che perforera' qualsiasi lattina :P) o anche di ceramica, Pbs finemente tritato e soda caustica, in proporzioni NaOH : PbS = 0.18 : 1.8 e aggiungere al totale 10% del peso risultate in acqua, e mescolare fino a che si ottiene una sorta di fango. attenzione, sviluppera' parecchio calore. dopo qualche minuto che ha smesso di generare calore, il composto sara' giallastro, a questo punto riscaldare il crogiolo tra 20 e 200 gradi, insomma, finche non evapora tutta l'umidita' e rimane secco. A questo punto, portare la miscela a 650° (io ho usato un bidone refrattario, ma qualsiasi fonte di calore + ambiente refrattario che possa raggiungere questa temperatura uniformemente va bene) e dopo un certo tot di tempo, si otterra' la "metallizzazione" ovvero abbastanza velocemente il composto diventera' la sua forma metallica.
Se non ho capito male, questo processo avviene per via della capacita' dello zolfo e dei solfati di cedere elettroni.

Vorrei postarvi le foto del materiale ottenuto, e' fantastico :D
Nel mio caso da 120 grammi di Pbs ho ricavato 85 grammi di piombo metallico, ma considerate anche che la lattina verso il pieno completamento del processo (evidentemente sviluppa anche calore di per se) si e' forata e ho spento :P

Saluti ragazzi!